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Adhesive Bonding Performance of Aerospace Materials Prepared With Alternative Solvents

by Scott M. Grendahl, Wai K. Chin,
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Adhesive Bonding Performance of Aerospace Materials Prepared With Alternative Solvents

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Abstract

The purpose of this work was to identify a suitable, environmentally friendly maintenance chemical to replace methyl ethyl ketone (MEK), a standard solvent currently utilized for adhesive bonding of metal substrates. MEK was used as a baseline reference maintenance chemical for this project. The effectiveness of three environmental friendly replacement candidate compounds were evaluated under the repair simulation guidelines of the Aeronautical Design Standard Performance Specification for Cleaners, Aqueous and Solvent, for Army Aircraft (U.S. Army Aviation and Missile Command. "Aeronautical Design Standard Performance Specification for Cleaners, Aqueous and Solvent, for Army Aircraft." ADS-61-PRF, Draft, Aviation Engineering Directorate, Redstone Arsenal, AL, 16 May 2000), and the Standard Test Method for Floating Roller Peel Resistance of Adhesives, ASTM-D3167-93 (American Society for Testing and Materials. "Standard Test Method for Floating Roller Peel Resistance of Adhesives." ASTM D3167-93, West Conshohocken, PA, 1993). Four metal substrates (AM-355 stainless steel, electroformed nickel plated steel, aluminum 7075-T6 bare, and titanium 6Al-4V) and four chemicals (identified as MEK, normalized propylbromide [NPB], Vertec Gold, and HFE 71DE) were utilized. The adhesive utilized in the layup of the test specimens was the two-part epoxy paste system—Dexter Hysol EA 9309.3NA. The results indicated that the best replacement candidates were Vertec Gold, an ethyl lactate-based cleaner, and HFE 71DE, a solvent-based cleaner.

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1. Introduction

One of the most difficult areas of overhaul and repair processing from which to remove hazardous chemicals is the area of adhesive bonding (AB). Great pains have been taken attempting to uncover alternative processing methods that preclude the use of these hazardous chemicals due to increased pressure from the Environmental Protection Agency (EPA) and the Occupational Safety and Health Administration (OSHA). With increased pressure to reduce these hazardous and environmentally unfriendly chemical cleaning agents, come new chemical alternatives and processes. These new alternatives must be evaluated from both a compatibility and a performance standpoint. The U.S. Army Aviation and Missile Command (AMCOM) requested that the U.S. Army Research Laboratory (ARL) perform experiments addressing the possible replacement of methyl ethyl ketone (MEK) in the adhesive bonding of metal substrates process. The objective of the present work was to evaluate three potential alternatives and assess their performance in comparison to a standard accepted chemical cleaning agent, MEK. The prospective alternates were normalized propyl bromide (NPB) (Hypersolve NPB from W.R. Grace), HFE 71DE (3M Novec Engineered Fluids), and Ethyl Lactate (Vertec Gold from Vertec BioSolvents). In theory, any residue remaining on the surfaces after final hand-wipe cleaning with the prospective solvents would be detrimental to the strength of the adhesive bond. The more residue remaining, or if a specific residue was incompatible with the epoxy paste adhesive, the weaker the bond strength would be when measured on an Instron mechanical testing load frame.

1.1 Testing Materials

The aviation materials utilized in the experiments were as follows:

- AM-355 Stainless Steel

  AM-355 is a semi-austenitic precipitation hardenable stainless steel. The material for this work was in the cold rolled and tempered condition (CRT) and in all cases was 0.014 in for the flexible adherend and 0.1 in for the rigid adherend.

- Electroformed Nickel-Coated 4130 Steel

  4130 sheet stock in 0.005 in for the flexible adherend and 0.032 in for the rigid adherend was utilized. Both sides were coated with electroformed nickel at 0.003–0.005 in.

- Titanium 6-4

  Titanium alloy with 6% aluminum and 4% vanadium was 0.025 in for the flexible adherend and 0.063 in for the rigid adherend.

- Aluminum 7075

  Bare (not anodized or conversion coated) aluminum alloy 7075 in the T6 heat-treat condition was 0.025 in for the flexible adherend and 0.063 in for the rigid adherend.

1.2 Testing Solutions

- NPB, Hypersolve NPB, manufactured by Great Lakes Chemical, West Lafayette, IN;
- HFE-71DE, 3M Novec Engineered Fluids, St. Paul, MN;
- Ethyl Lactate, Vertec Gold, Vertec BioSolvents, Mt. Prospect, IL; and
- MEK.

1.3 Standard Contaminant

The standard contaminant was two parts by weight of hydraulic fluid (MIL-PRF-83282[1] or equivalent), one part by weight lubricating grease (MIL-PRF-81322[2] or equivalent), and one-tenth by weight carbon black. The mixture was applied uniformly with a paintbrush on each metal panel surface. The panels were then baked for two hr at 55 °C (130 °F), removed from the oven, and cooled.

2. Adhesive Bonding Procedure

The procedures outlined within Aeronautical Design Standard 61[3] (ADS-61-PRF) were followed. The AB performance testing evaluates whether or not each test solution provides a better AB surface than the control solution (MEK). In all cases, the test panels were contaminated with the standard contaminant and baked at 130 °F for two hr. The test solutions and the control solution, MEK, were then utilized to hand wipe clean the respective panels. The panels were then scuffed with an orbital sander. Aluminum panels were scuffed with

[1]U.S. Naval Air Systems Command. "Hydraulic Fluid, Fire Resistant, Synthetic Hydrocarbon Base, Metric, NATO Code Number H-537." MIL-PRF-83282, Lakehurst, NJ, December 1997.

[2]U.S. Naval Air Systems Command. "Grease, Aircraft, General Purpose, Wide Temperature Range." MIL-PRF-81322, Lakehurst, NJ, July 1998.

[3]U.S. Army Aviation and Missile Command. "Aeronautical Design Standard Performance Specification for Cleaners, Aqueous and Solvent, for Army Aircraft." ADS-61-PRF, Draft, Aviation Engineering Directorate, Redstone Arsenal, AL, 16 May 2000.

180-grit, titanium with 120-grit, and nickel and AM-355 steel with 80-grit sanding discs. All panels were subsequently recleaned with the same respective hand-wipe cleaner as was previously performed. Specimen panel sets were bonded and cured with Dexter Hysol 9309.3NA paste adhesive in accordance with American Society for Testing and Materials (ASTM)-D3167.[4] The panel sets were then cut, via water jet cutting, into 0.5-in strip specimens for floating roller peel (FRP) testing. Lap shear panel sets were also fabricated in a similar fashion for aluminum 7075 and titanium 6-4. Representative FRP specimens from each group of bonded panel sets were then tested in accordance with ASTM-D3167 at both room temperature (RT), and 180 °F after 30 days in 95% humidity at 180 °F. The average bond line thickness was approximately 0.007 in. The glass beads within the 9309.3NA paste adhesive were 0.005 in. The panels for the bonded sets were 10 × 3 × thickness (inches) for the flexible adherend and 8 × 3 × thickness (inches) for the rigid adherend. The bonding strength, in pounds, was recorded. The lap shear tests were conducted at RT and the fracture load, in pounds, was recorded. The accept/reject criteria were that the test solutions provide better or equivalent adhesion when compared with the control solution, MEK. The FRP testing was performed according to specifications set forth in ASTM-D3167. An Instron Model 5500 Universal Testing Machine was used. The crosshead speed on the Instron was set at the rate of 6 in (152 mm)/min.

3. Adhesive Bonding Results

The AB performance of the prospective test solutions was evaluated by comparing the effects of the residue remaining on the surface by contrasting the adhesion values recorded with a control solution, MEK. Table 1 presents the average load value of the FRP tests for each group of specimens conditions performed at RT and 180 °F. Additionally, the pounds per lineal inch (PLI) are provided. The results that are considered equal to or better than the control group are highlighted. There existed considerable scatter in the data consistent with typical floating roller peel test results. However, clear differences and trends can be observed. Figures 1–4 graphically depict the bonding results of the prospective test solutions and MEK at RT on AM-355, electroformed nickel, titanium and aluminum, respectively. The graphs present the average load from each specimen at each condition along with the average load for all the specimens of the same condition. The data from similar conditions are presented as hues of the same color. MEK cleaned specimens have red hues, NPB cleaned specimens have green hues, HFE 71DE cleaned specimens have blue hues, and ethyl lactate cleaned specimens have yellow hues.

[4]American Society for Testing and Materials. "Standard Test Method for Floating Roller Peel Resistance of Adhesives." ASTM-D3167, West Conshohocken, PA, 1993.

Table 1. Results of the FRP testing at varying conditions.

Material	Testing Solution	Testing Conditions	Average FRP Load (lb)	Average FRP Load (PLI)	Failure Type
AM-355	MEK	RT	0.98	1.96	Adhesive
AM-355	NPB	RT	1.08	2.16	Adhesive
AM-355	HFE 71 DE	RT	0.71	1.42	Adhesive
AM-355	Ethyl Lactate	RT	1.18	2.36	Adhesive
AM-355	MEK	180 °F	3.30	6.6	Adhesive
AM-355	NPB	180 °F	4.05	8.1	Adhesive
AM-355	HFE 71 DE	180 °F	2.90	5.8	Adhesive
AM-355	Ethyl Lactate	180 °F	4.20	8.4	Adhesive
Electroformed Ni	MEK	RT	0.59	1.18	Adhesive
Electroformed Ni	NPB	RT	0.49	0.98	Adhesive
Electroformed Ni	HFE 71 DE	RT	0.68	1.36	Adhesive
Electroformed Ni	Ethyl Lactate	RT	0.90	1.8	Adhesive
Electroformed Ni	MEK	180 °F	1.11	2.22	Adhesive
Electroformed Ni	NPB	180 °F	0.42	0.84	Adhesive
Electroformed Ni	HFE 71 DE	180 °F	1.27	2.54	Adhesive
Electroformed Ni	Ethyl Lactate	180 °F	1.56	3.12	Adhesive
Titanium 6-4	MEK	RT	0.75	1.5	Adhesive
Titanium 6-4	NPB	RT	0.45	0.9	Adhesive
Titanium 6-4	HFE 71 DE	RT	0.55	1.1	Adhesive
Titanium 6-4	Ethyl Lactate	RT	2.90	5.8	Adhesive
Titanium 6-4	MEK	180 °F	1.63	3.26	Adhesive
Titanium 6-4	NPB	180 °F	0.83	1.66	Adhesive
Titanium 6-4	HFE 71 DE	180 °F	0.90	1.8	Adhesive
Titanium 6-4	Ethyl Lactate	180 °F	1.16	2.32	Adhesive
Aluminum 7075	MEK	RT	1.45	2.9	Adhesive
Aluminum 7075	NPB	RT	0.60	1.2	Adhesive
Aluminum 7075	HFE 71 DE	RT	0.95	1.9	Adhesive
Aluminum 7075	Ethyl Lactate	RT	1.75	3.5	Adhesive
Aluminum 7075	MEK	180 °F	0.80	1.6	Adhesive
Aluminum 7075	NPB	180 °F	0.78	1.56	Adhesive
Aluminum 7075	HFE 71 DE	180 °F	1.43	2.86	Adhesive
Aluminum 7075	Ethyl Lactate	180 °F	3.10	6.2	Adhesive

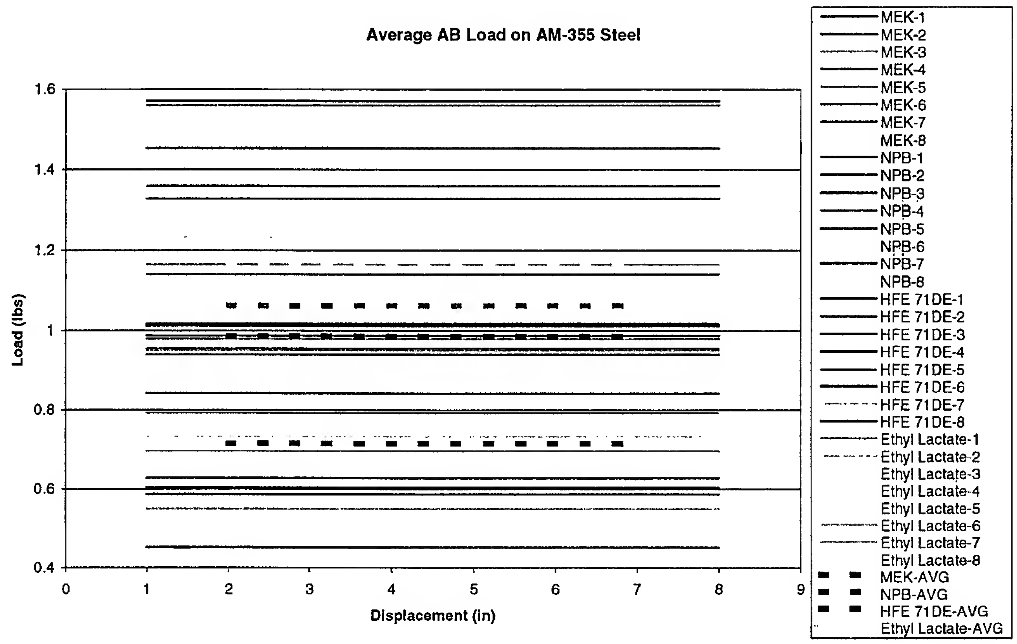

Figure 1. Average AB load on cleaned AM-355 steel, tested at RT.

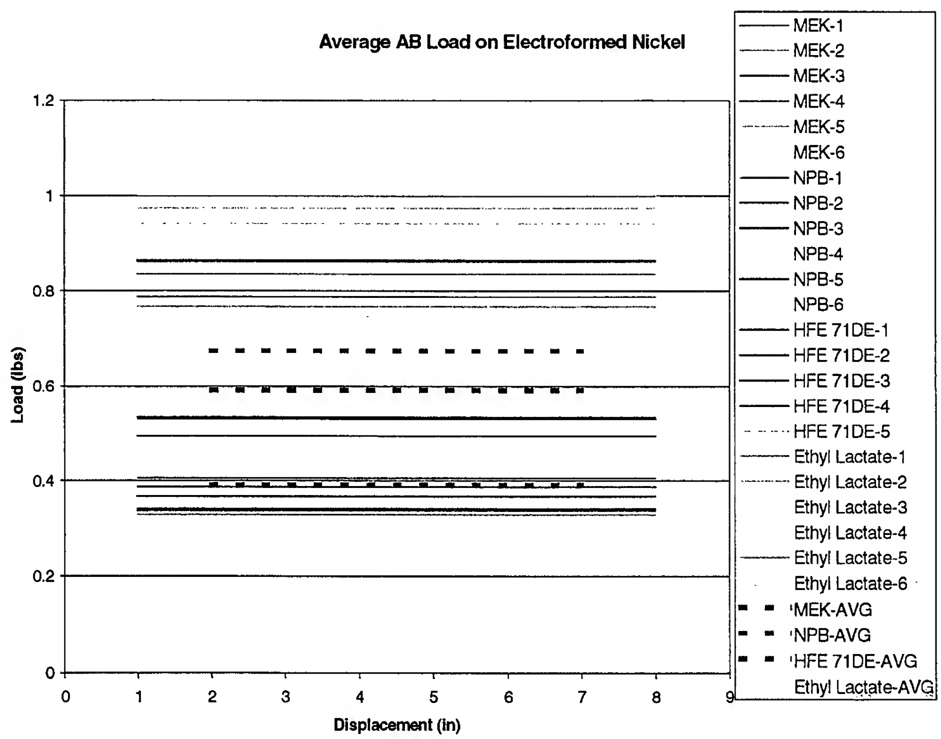

Figure 2. Average AB load on cleaned electroformed nickel, tested at RT.

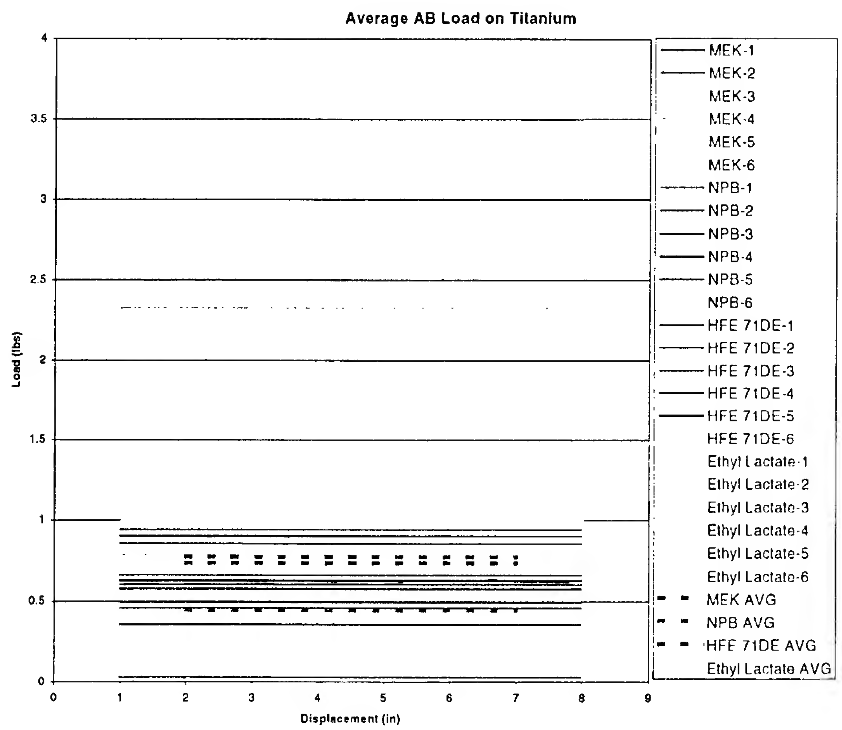

Figure 3. Average AB load on cleaned titanium, tested at RT.

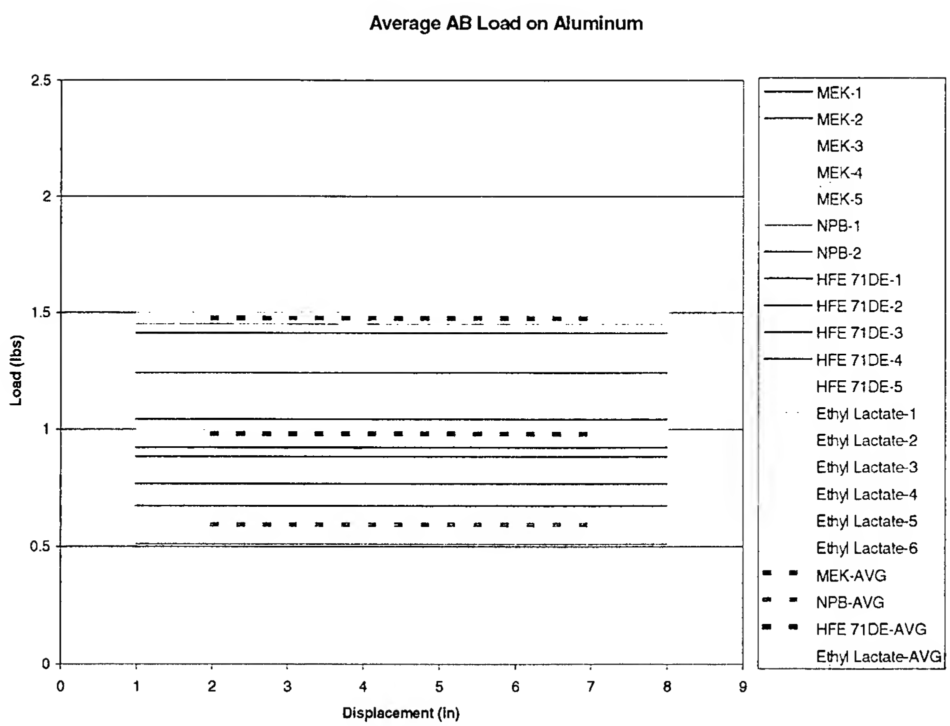

Figure 4. Average AB load on cleaned aluminum, tested at RT.

Figures 5–8 graphically depict the bonding results of the prospective test solutions and MEK tested at 180 °F on AM-355, electroformed nickel, titanium and aluminum, respectively. The individual results from each panel for the RT tests and the 180 °F testing are included as Appendix A and Appendix B, respectively.

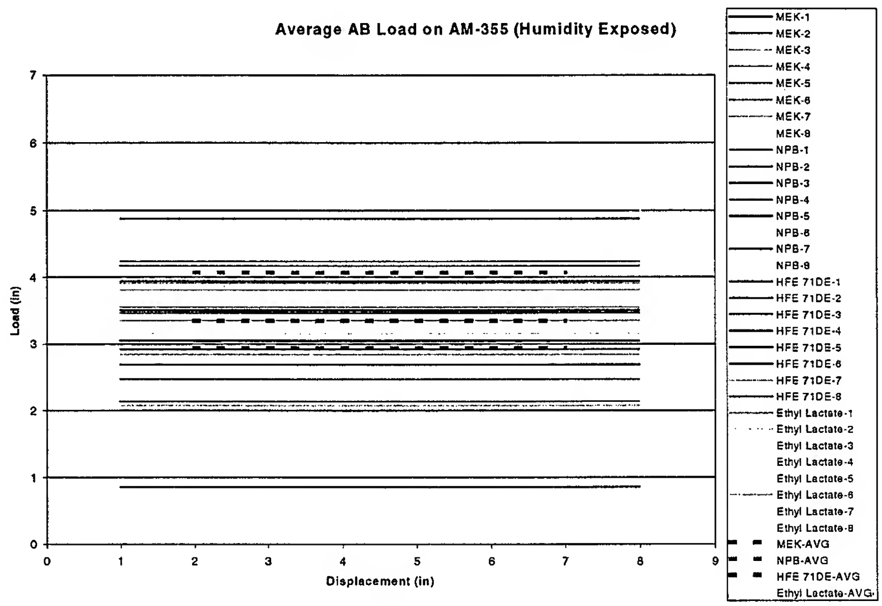

Figure 5. Average AB load on cleaned AM-355 steel, tested at 180 °F.

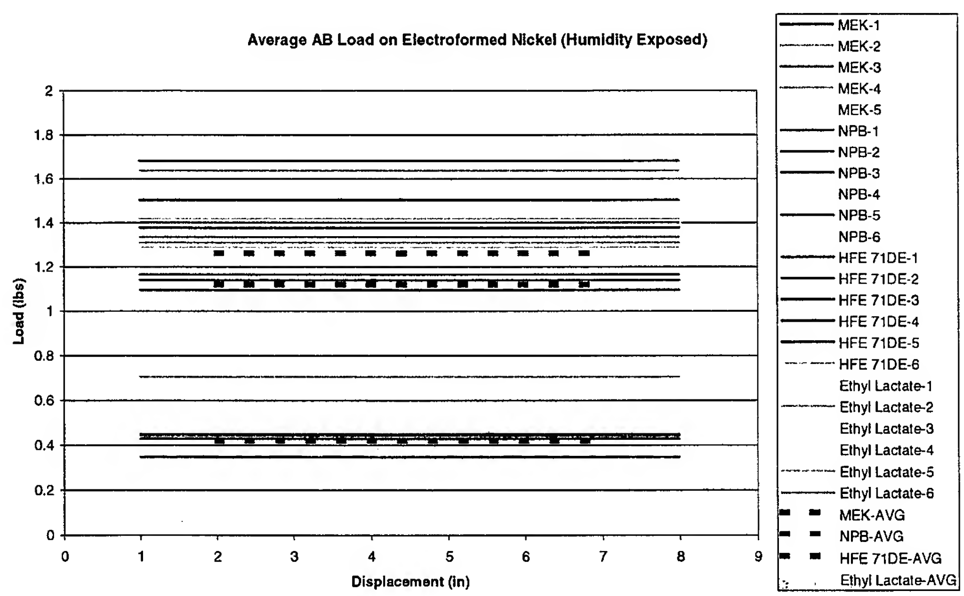

Figure 6. Average AB load on cleaned electroformed nickel, tested at 180 °F.

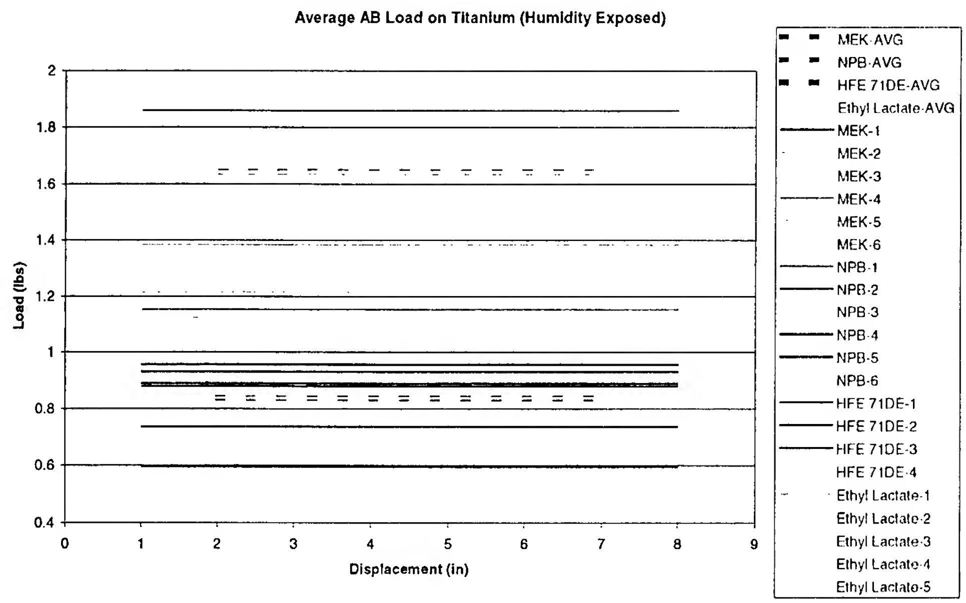

Figure 7. Average AB load on cleaned titanium, tested at 180 °F.

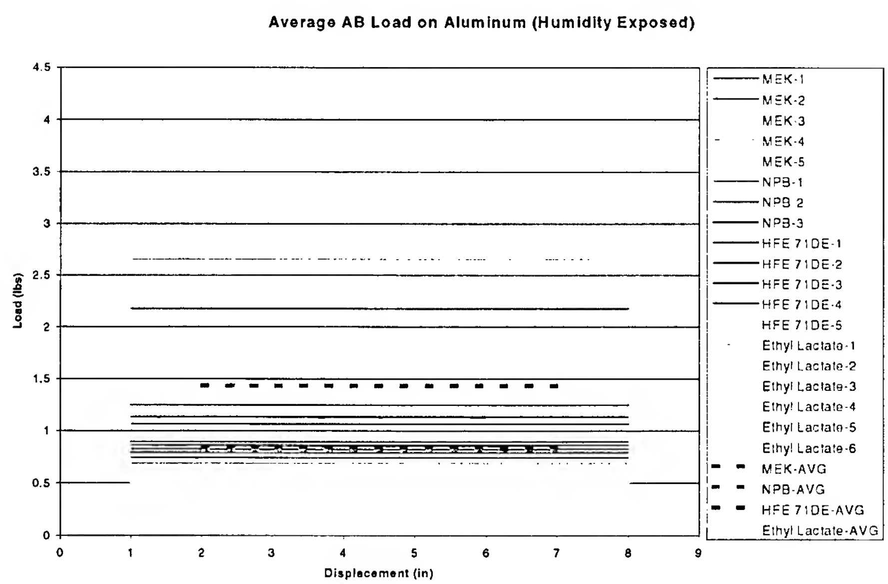

Figure 8. Average AB load on cleaned aluminum, tested at 180 °F.

The graphs clearly demonstrate that the ethyl lactate was the best cleaning solution for preparing the surfaces for bonding. The HFE 71 DE appears to satisfactorily prepare the surface when compared to the control cleaner, MEK. These results compared favorably with those from the preliminary lap shear

testing. The lap shear test results are presented in Table 2. The ethyl lactate group also proved to be the best performer under this testing schedule.

Table 2. Results of the AB lap shear testing.

Material	Testing Solution	Average Load at Fracture (lb)
Aluminum 7075	MEK	1877
Aluminum 7075	NPB	1794
Aluminum 7075	HFE 71DE	1741
Aluminum 7075	Ethyl Lactate	1907
Titanium 6-4	MEK	1945
Titanium 6-4	NPB	1869
Titanium 6-4	HFE 71DE	1889
Titanium 6-4	Ethyl Lactate	1961

4. Conclusion

The overall bonding levels were low, indicative of the repair processing simulation. It is believed that the data generated within this report is indicative of the performance of the repair processing at the maintenance facilities. The ethyl lactate cleaning agent, Vertec Gold, outperformed the other cleaning agents in all but one instance. It clearly demonstrated above-average ability to adequately prepare the surfaces to be bonded. This cleaning agent also had considerably longer evaporation times and was forced dried with hot air. HFE 71DE performed adequately in most cases. It is believed that the HFE 71DE cleaner could replace MEK in the AB processing of the subject materials.

5. Discussion

It is difficult to believe that the low adhesion levels achieved under this testing program replicate the actual bonding strengths of the maintenance repair work. However, the ADS-61 was precisely followed with respect to bonding and curing. It is believed that a more roughened surface profile (resulting from coarser grit sanding discs) would yield more consistent results and higher bonding strengths. The adhesive utilized is also becoming outdated. It is

difficult to believe that it is still widely utilized. It is extremely difficult to separate out performance when the data is overlapping and, in general, relatively low in value. Although the ethyl lactate cleaner seems to clearly be the best performer under this protocol, alternate surface preparation might yield more convincing results regarding the other solutions tested. The failure, in every case, was adhesive as opposed to cohesive, between the adhesive and the rigid adherend or between the adhesive and the flexible adherend. Usually, good bonding is evidenced by some amount of cohesive failure of the adhesive itself.

The ADS-61 should be updated to reflect that not all materials should be 0.025 and 0.063 in thick. The steel, AM-355, and the electroformed nickel with a steel substrate are too stiff to undergo this testing with 0.025 in as the thickness of the flexible adherend. The thickness utilized within this report should be viewed as guidelines for the correct thickness. It should be noted, however, that the nickel and the AM-355 were still very stiff even at the thickness ranges utilized in this report, and thinner substrates should be evaluated.

Appendix A. Room Temperature (RT) Testing

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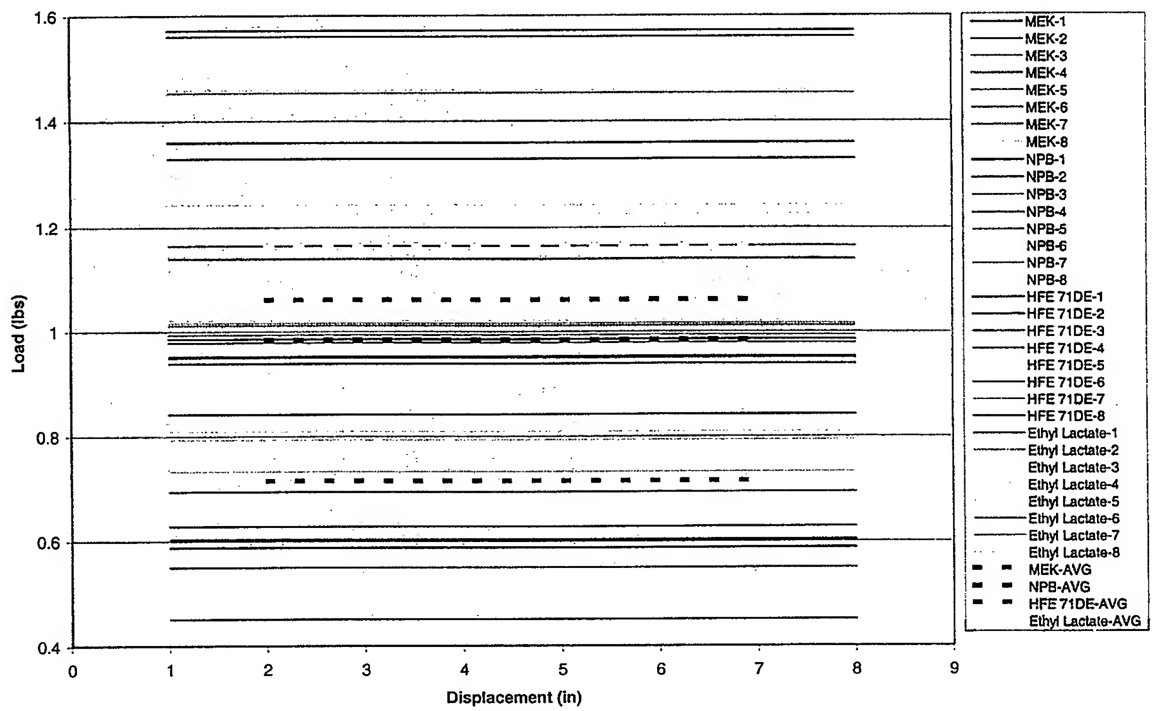

Figure A-1. Average AB load on AM-355 steel.

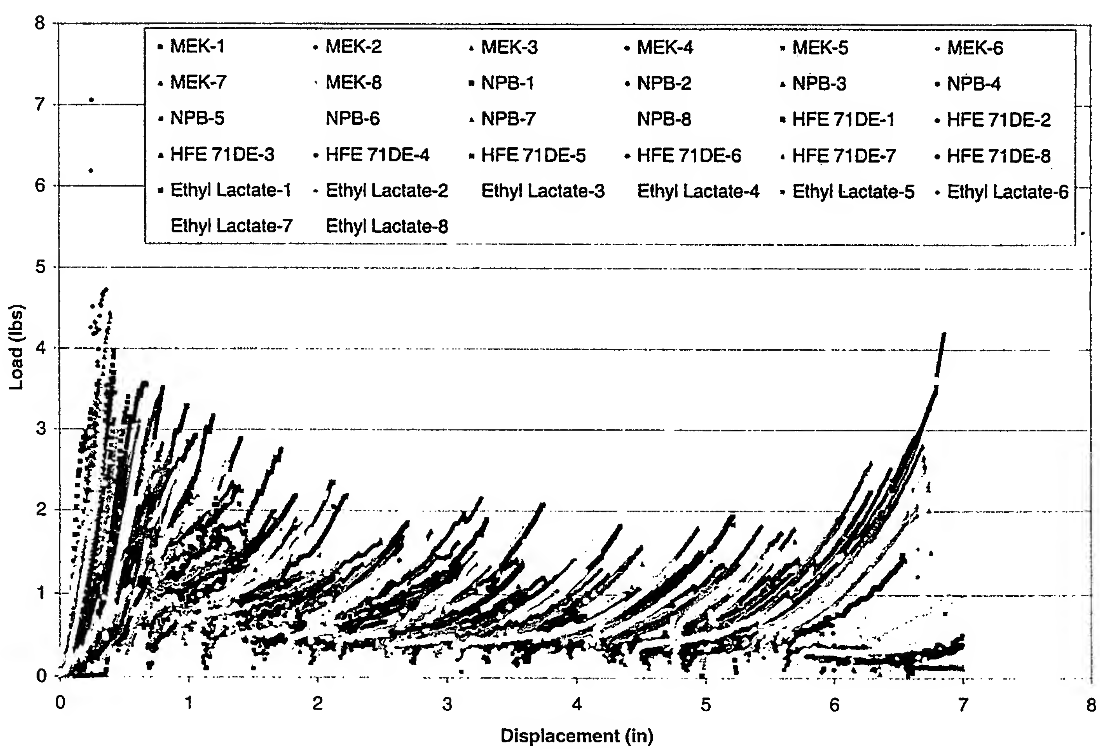

Figure A-2. AB performance of cleaned AM-355.

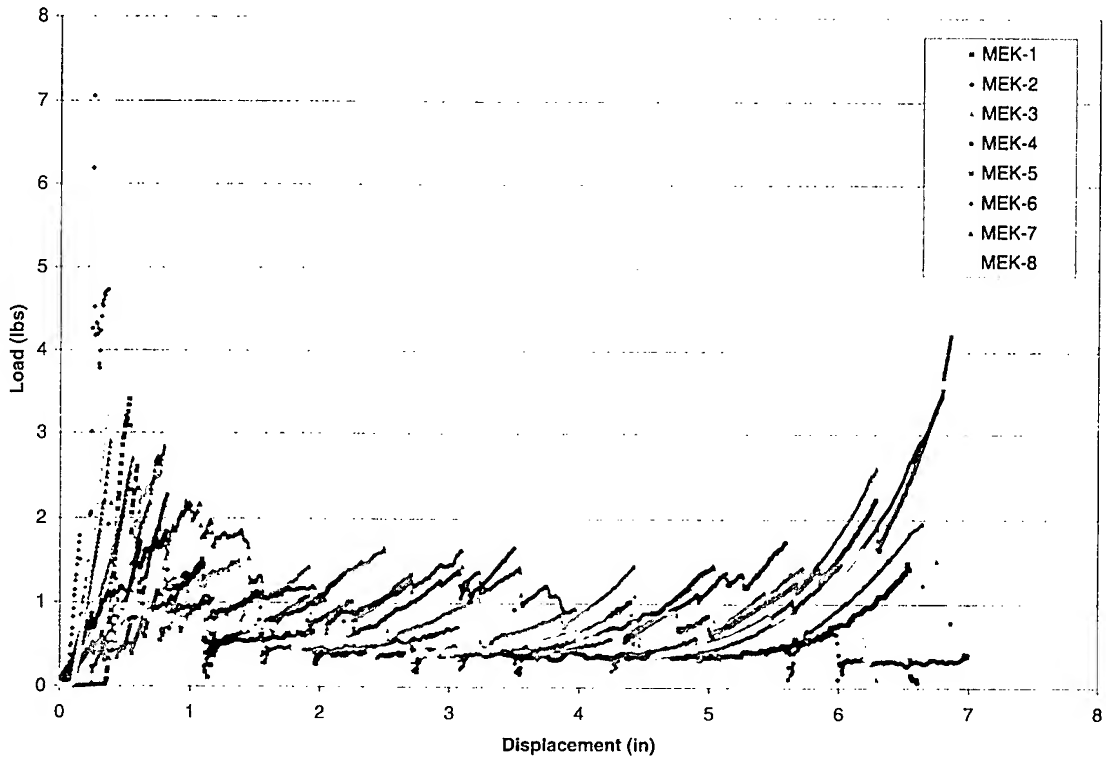

Figure A-3. MEK cleaned AM-355.

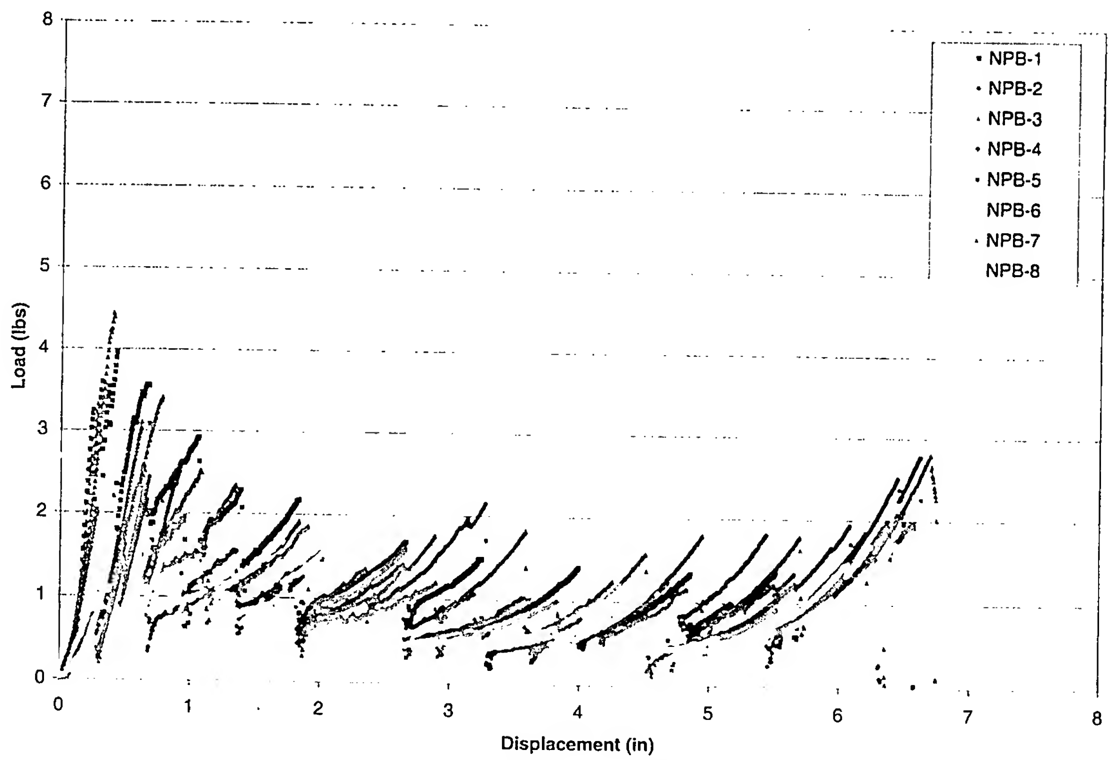

Figure A-4. NPB cleaned AM-355.

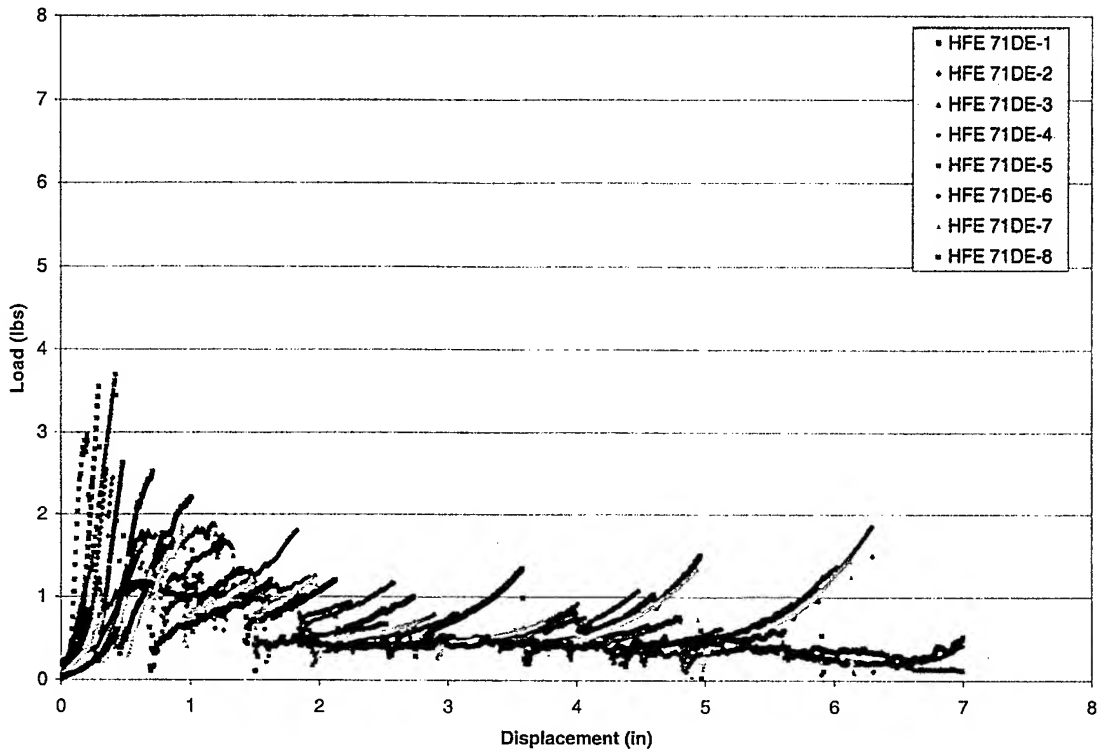

Figure A-5. HFE 71DE cleaned AM-355.

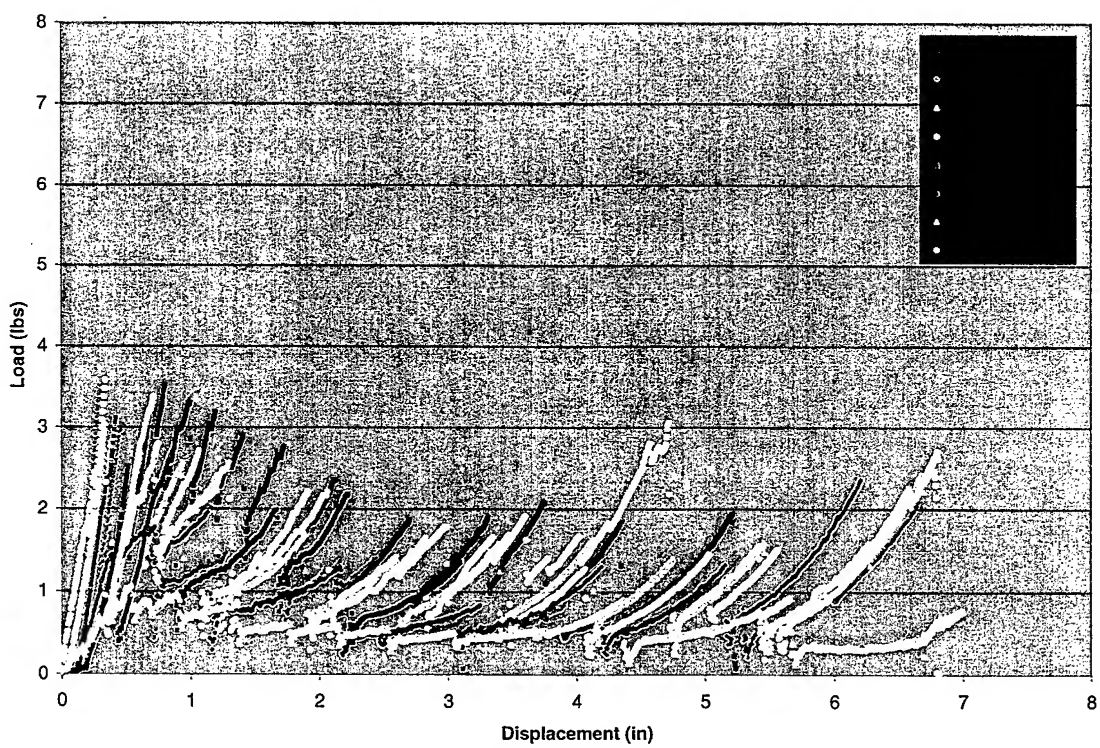

Figure A-6. Ethyl lactate cleaned AM-355.

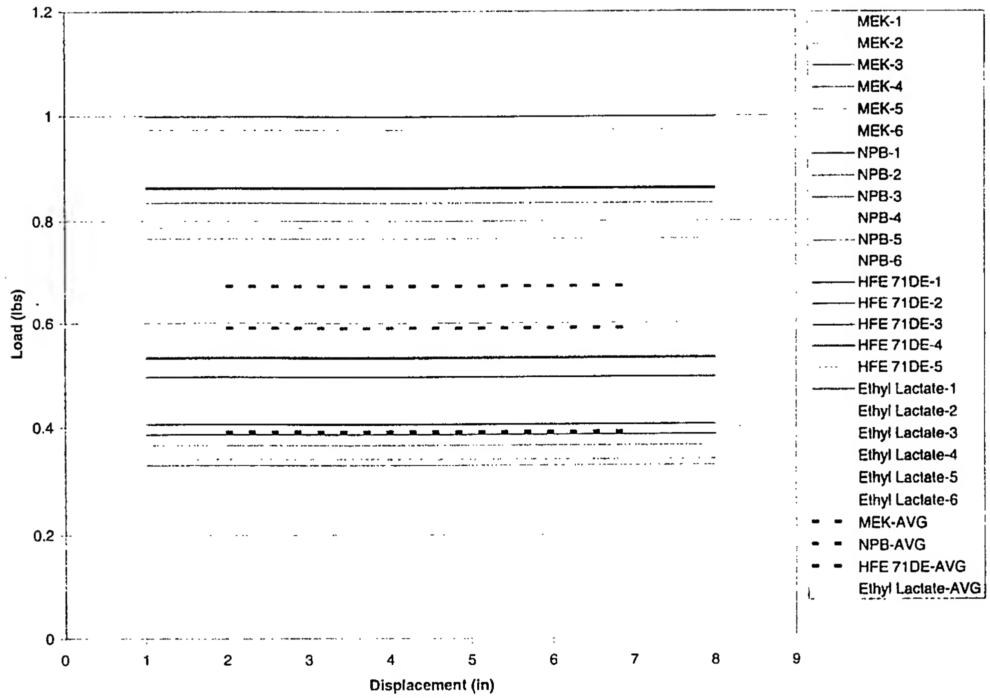

Figure A-7. Average AB load on electroformed nickel.

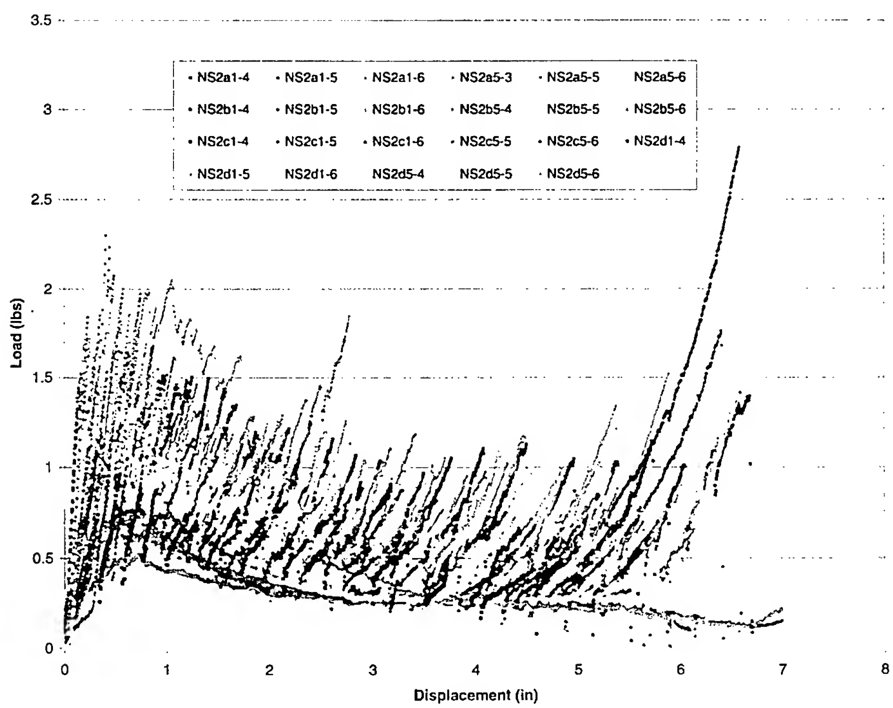

Figure A-8. Cleaned electroformed nickel.

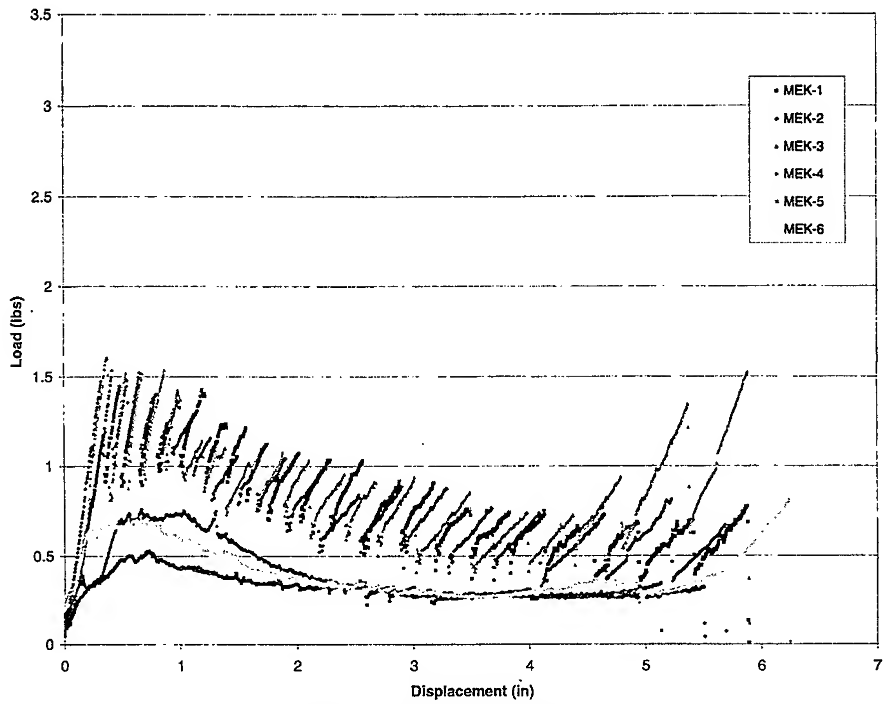

Figure A-9. MEK cleaned electroformed nickel.

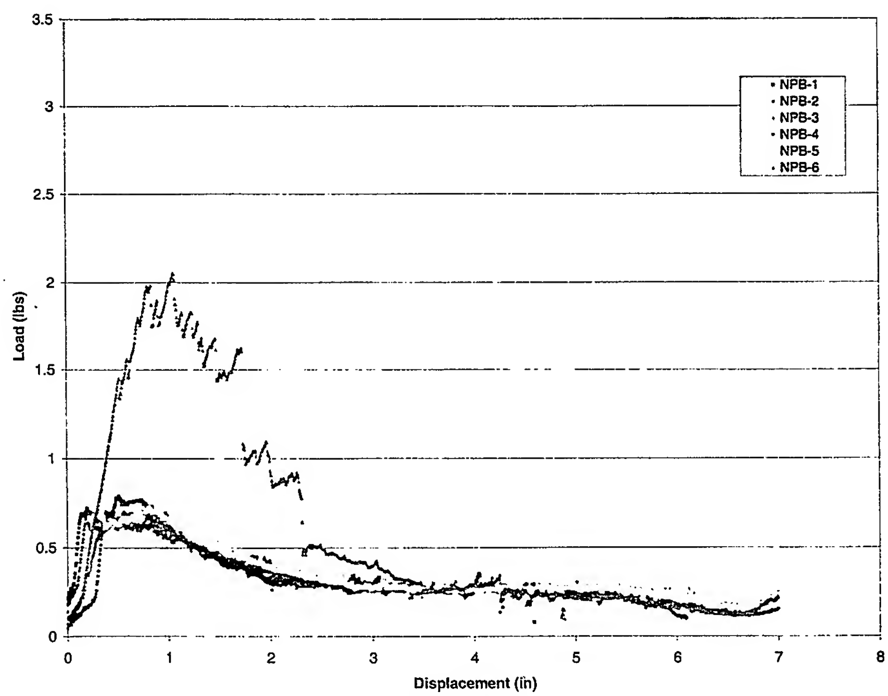

Figure A-10. NPB cleaned electroformed nickel.

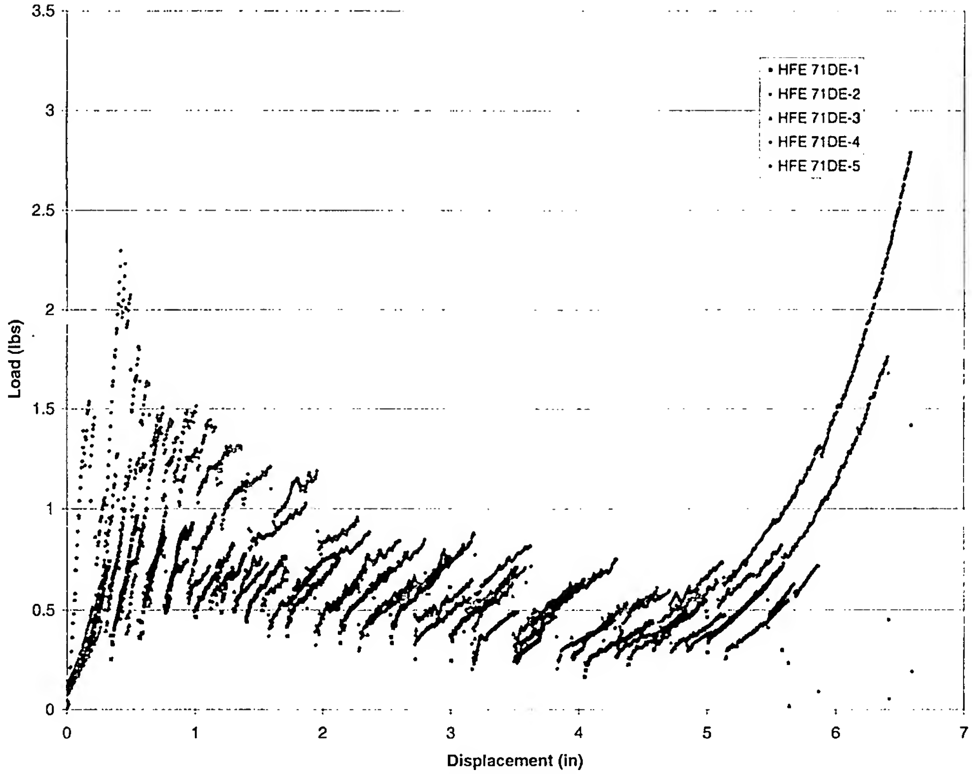

Figure A-11. HFE 71DE cleaned electroformed nickel.

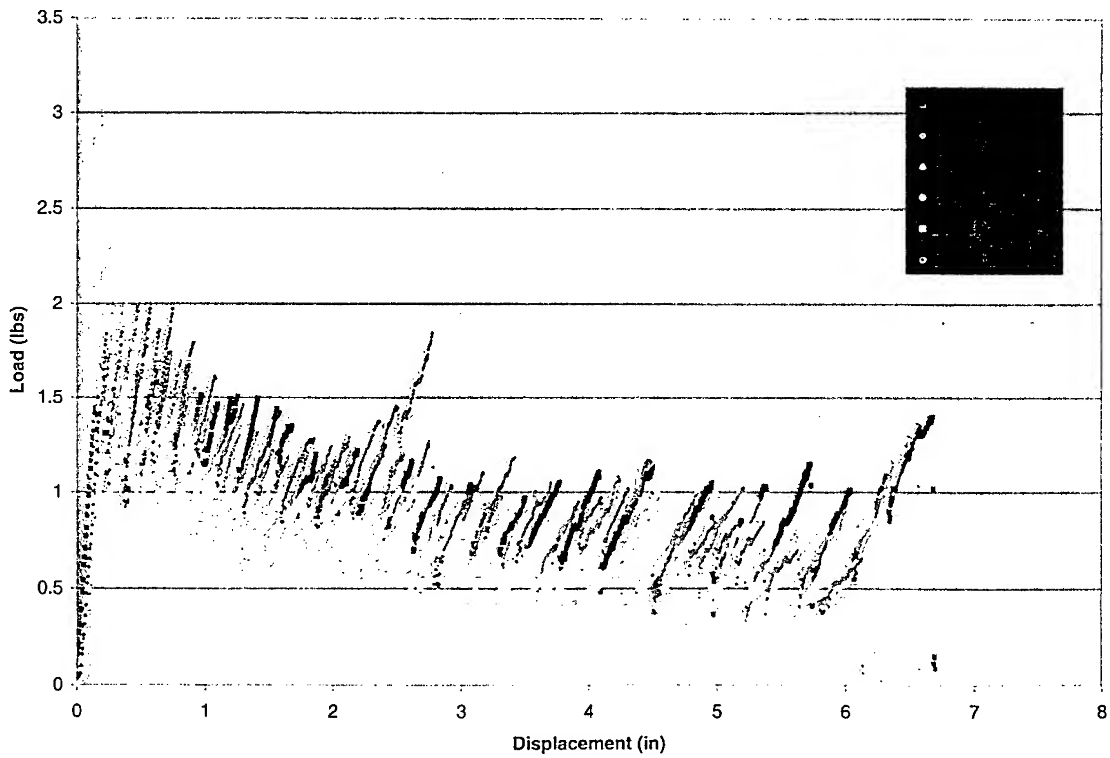

Figure A-12. Ethyl lactate cleaned electroformed nickel.

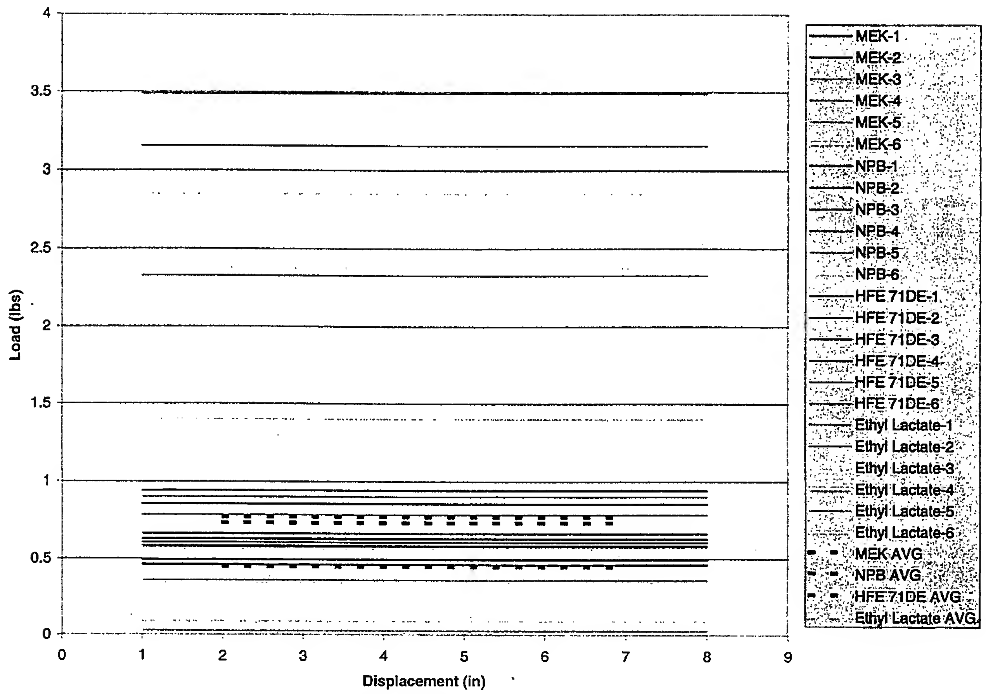

Figure A-13. Average AB load on titanium.

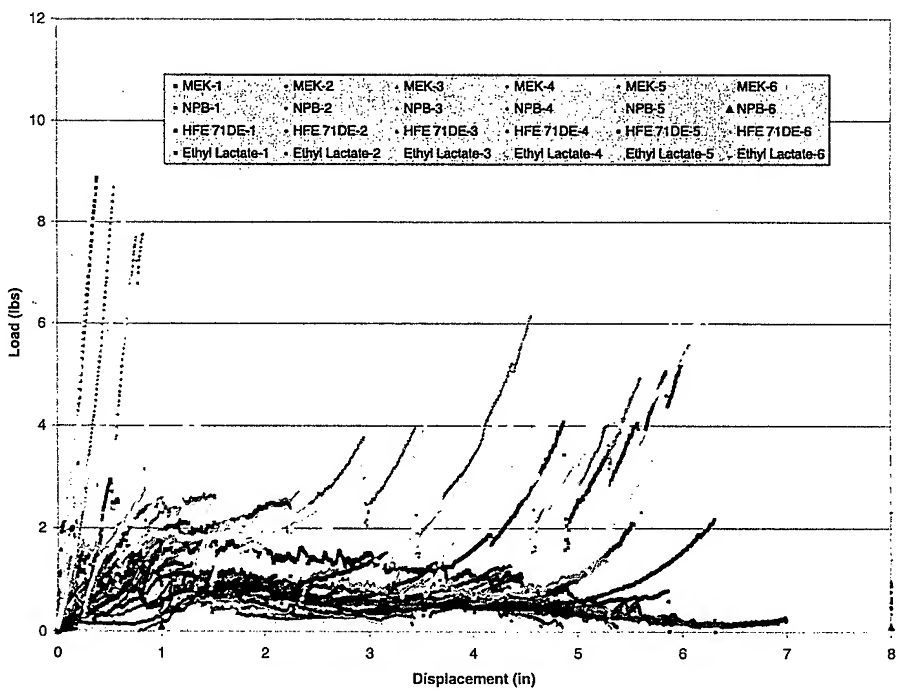

Figure A-14. AB performance of cleaned titanium.

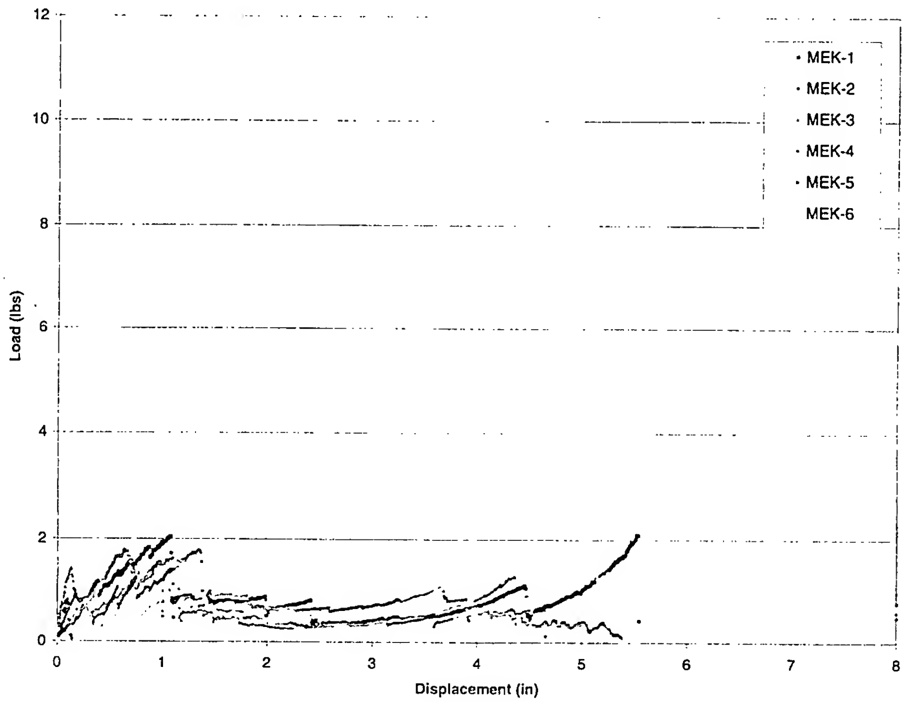

Figure A-15. MEK cleaned titanium.

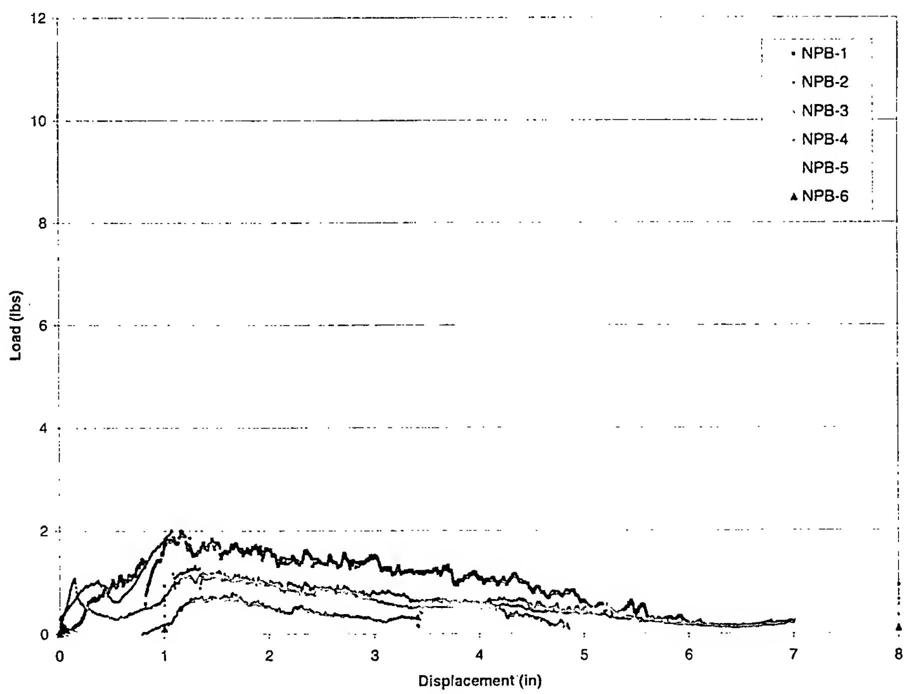

Figure A-16. NPB cleaned titanium.

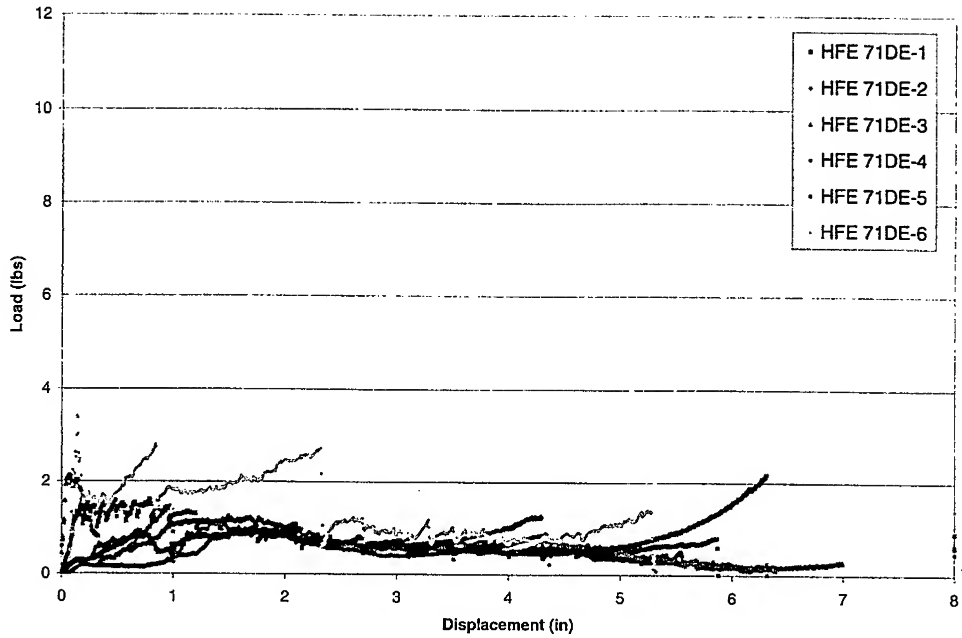

Figure A-17. HFE 71DE cleaned titanium.

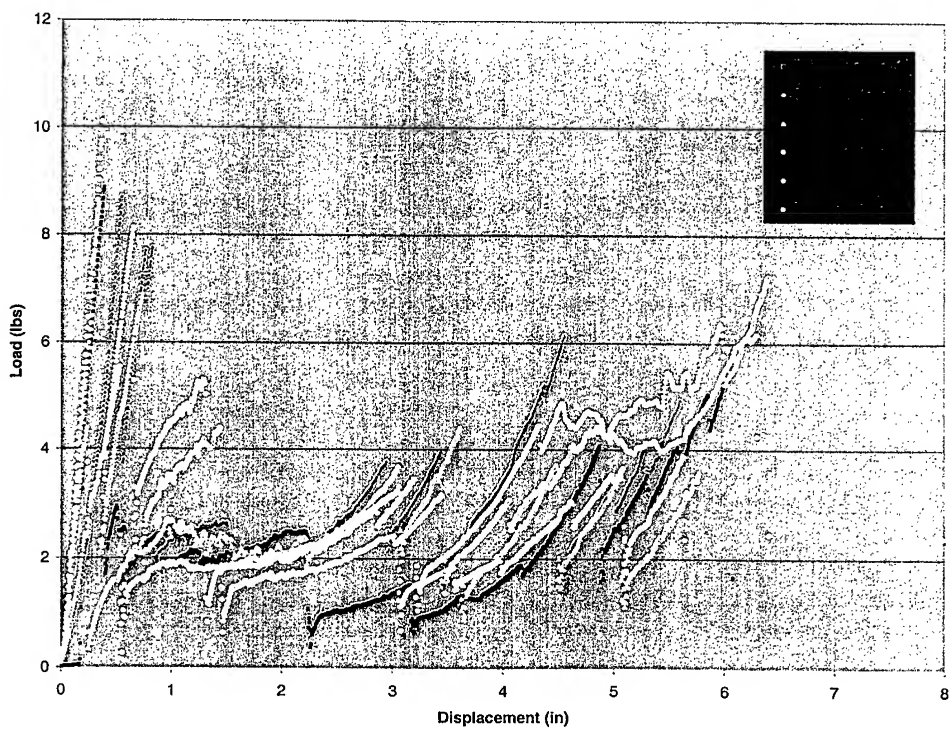

Figure A-18. Ethyl lactate cleaned titanium.

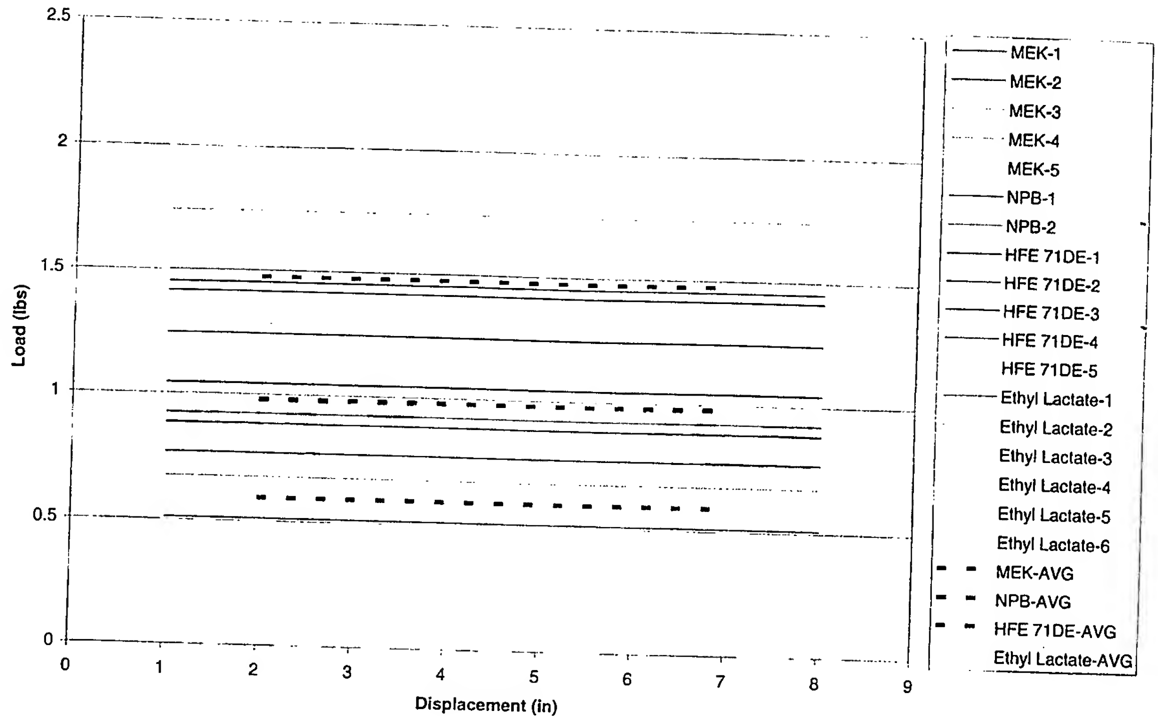

Figure A-19. Average AB load on aluminum.

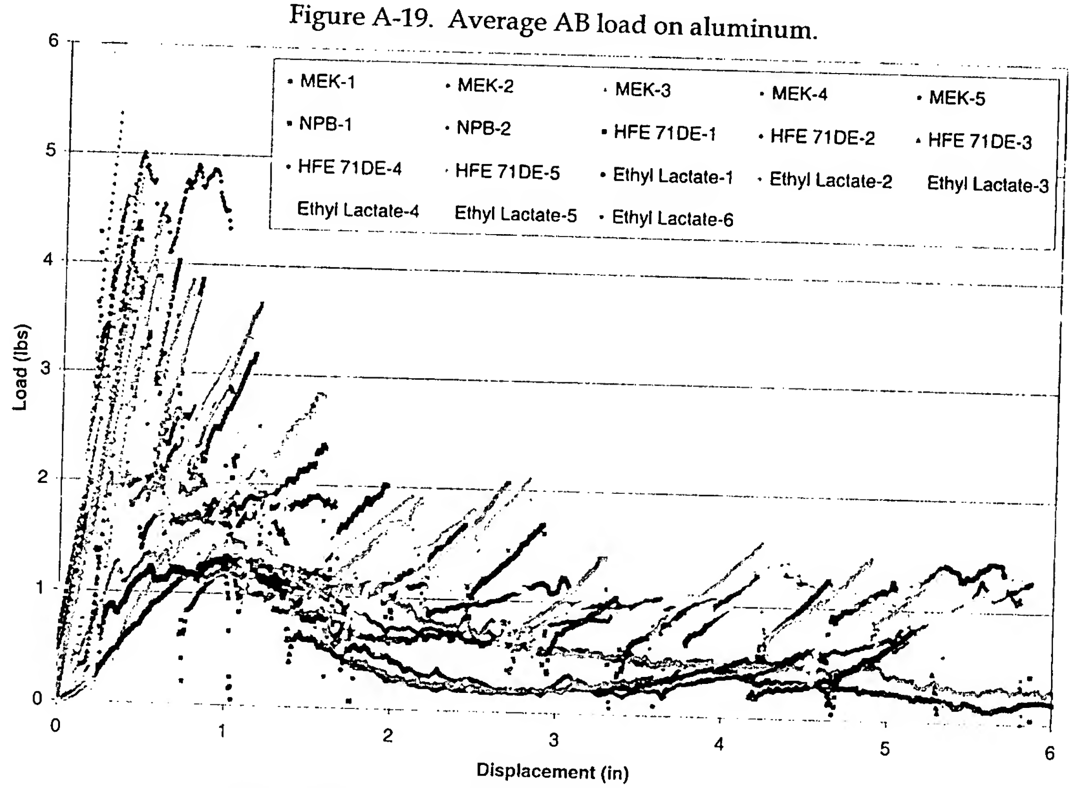

Figure A-20. AB performance of cleaned aluminum.

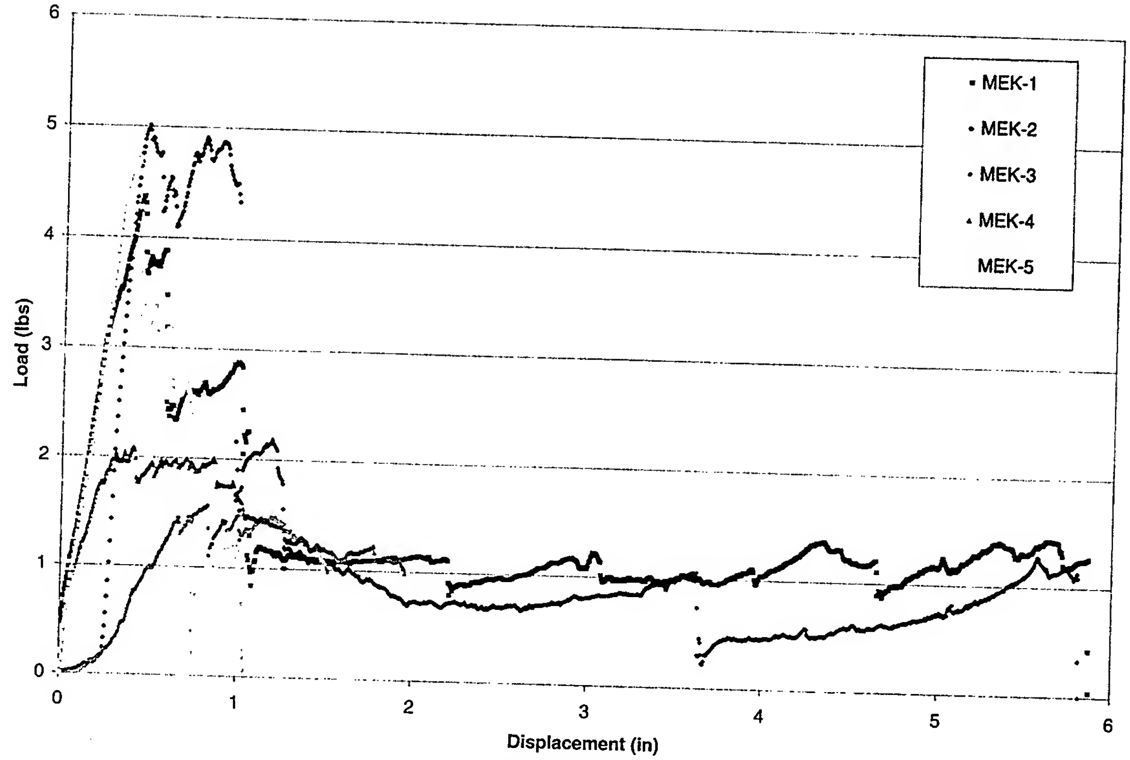

Figure A-21. MEK cleaned aluminum.

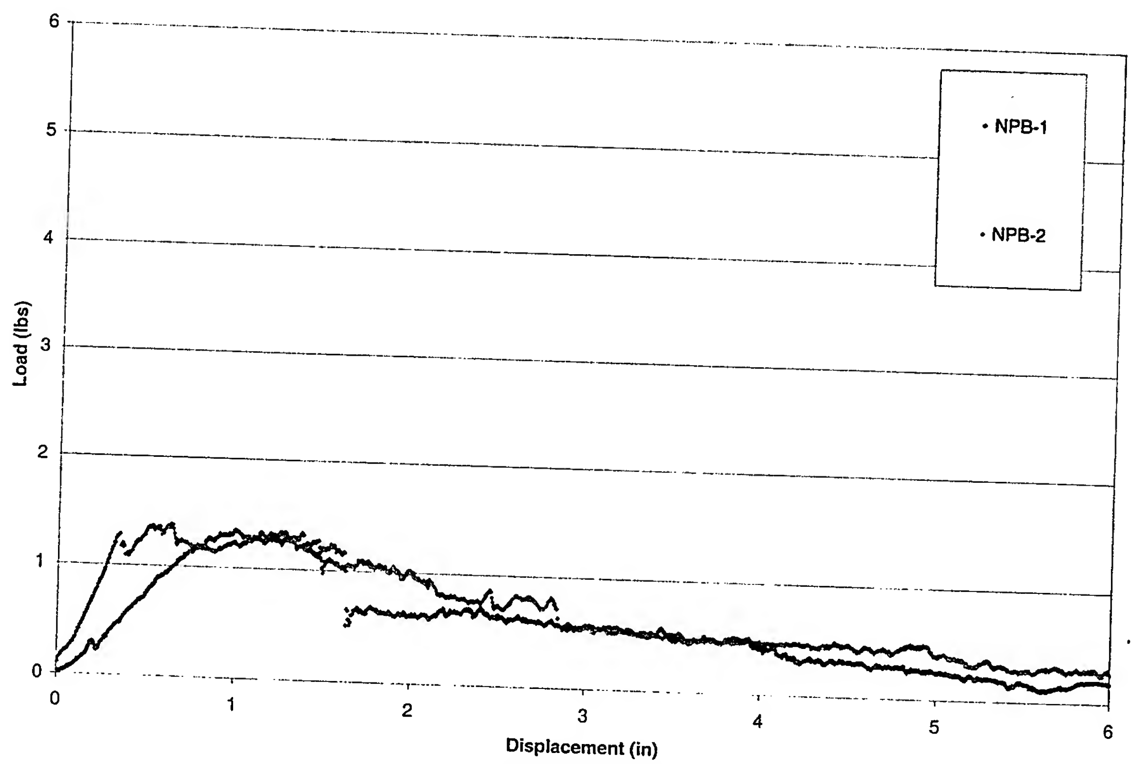

Figure A-22. NPB cleaned aluminum.

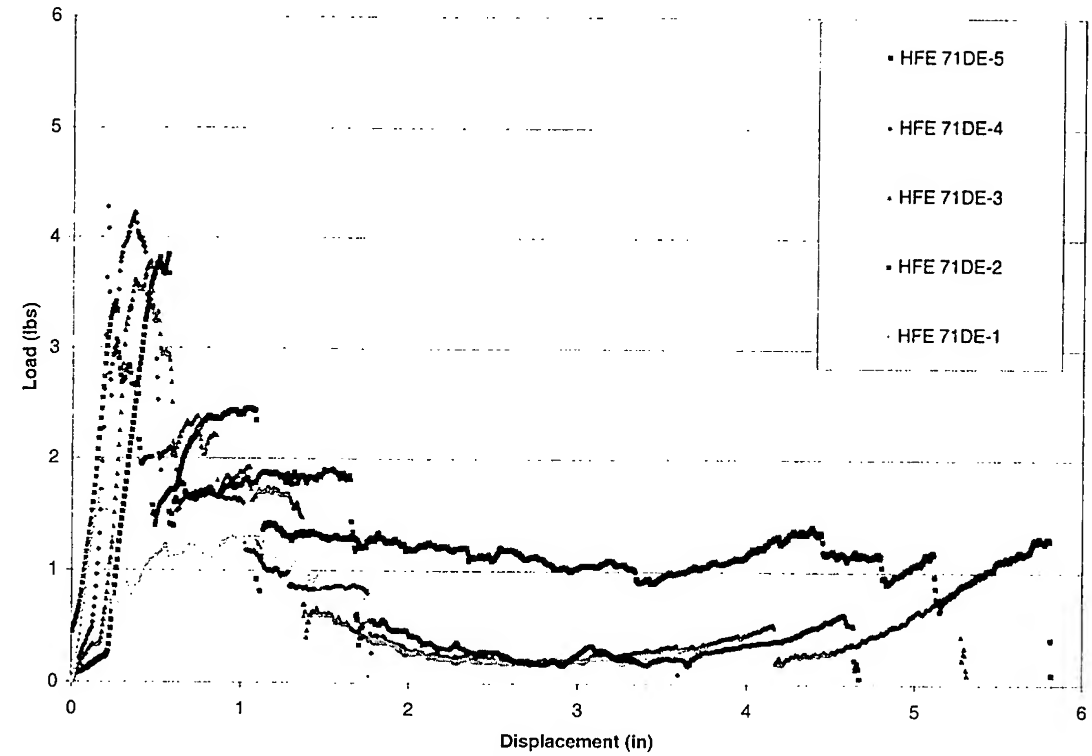

Figure A-23. HFE 71DE cleaned aluminum.

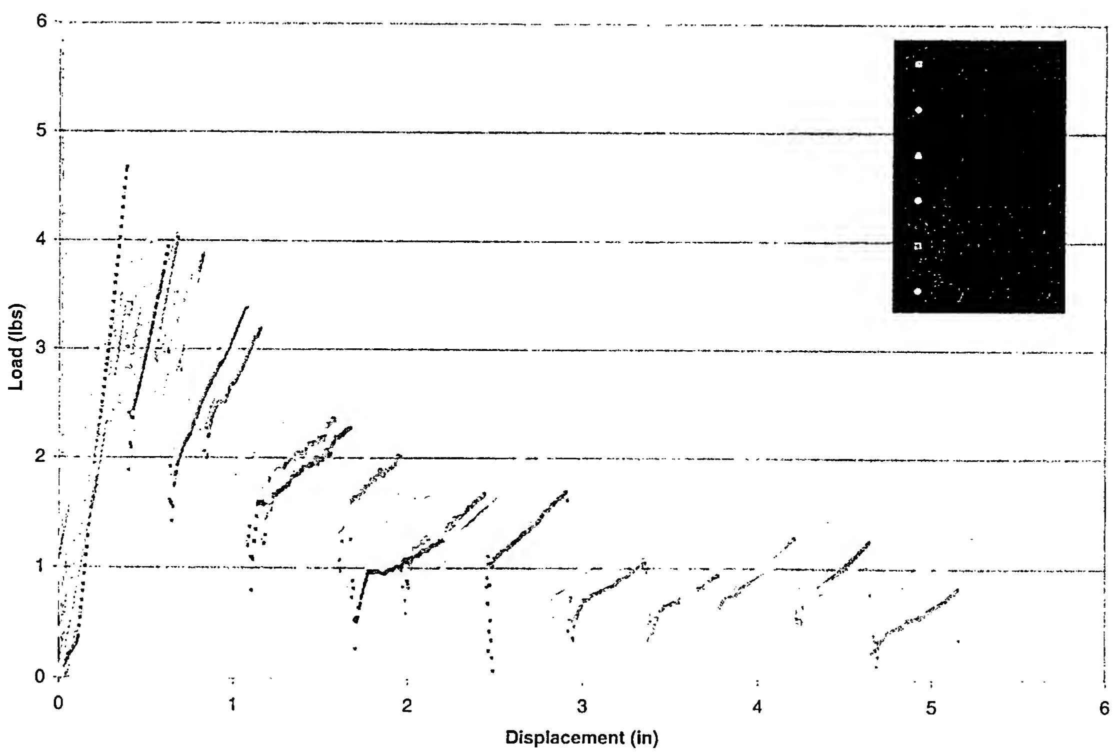

Figure A-24. Ethyl lactate cleaned aluminum.

Appendix B. 180 °F Testing

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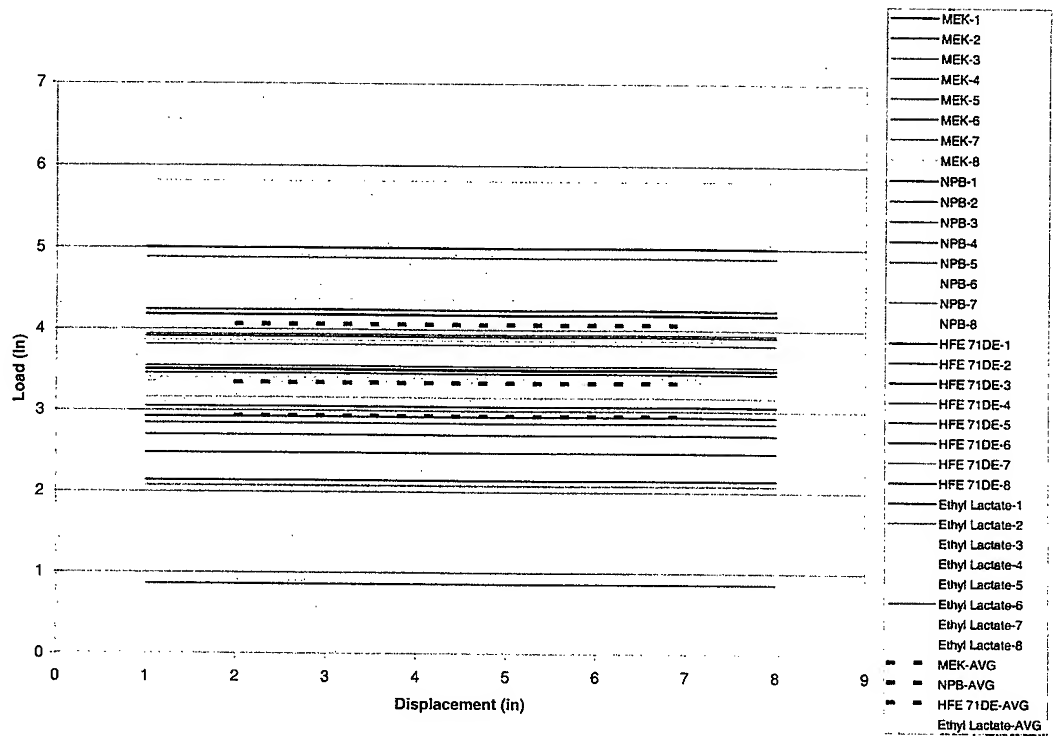

Figure B-1. Average AB loan on AM-355 (humidity exposed).

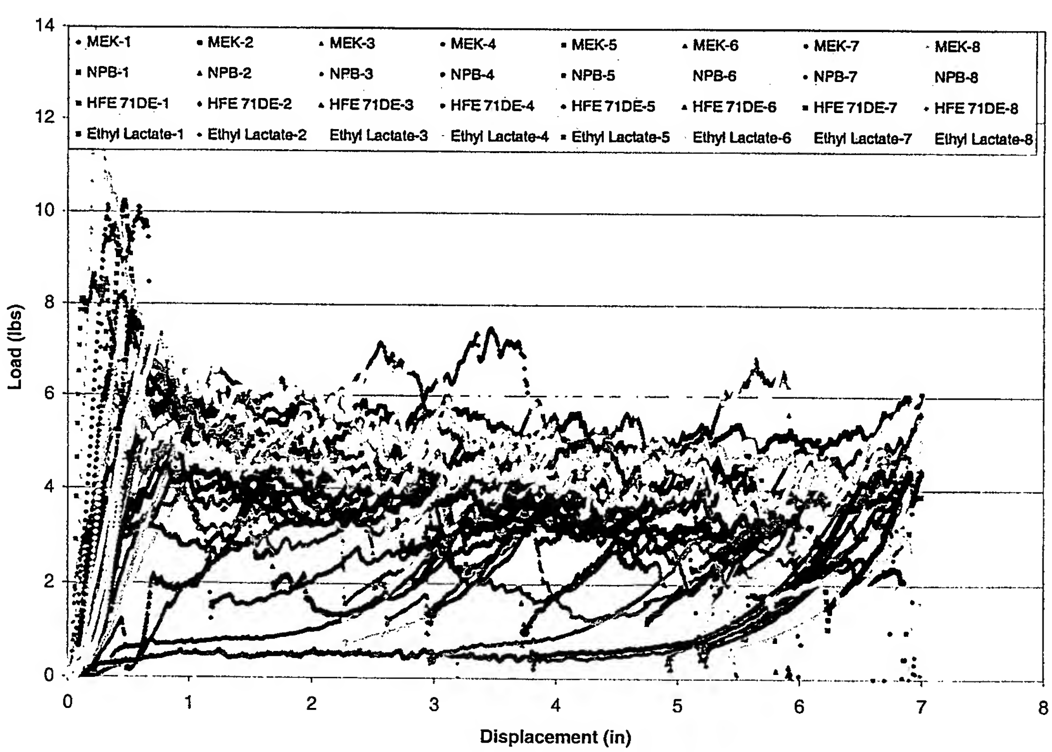

Figure B-2. AB performance of cleaned AM-355 (humidity exposed).

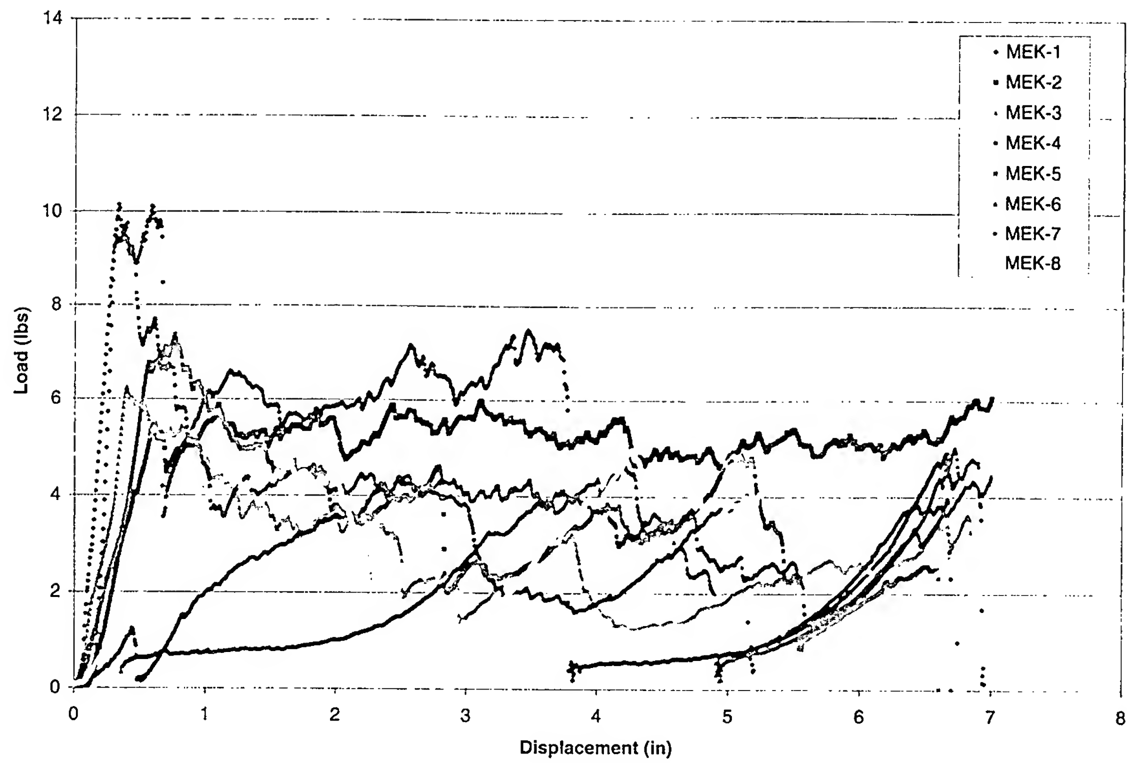

Figure B-3. MEK cleaned AM-355 (humidity exposed).

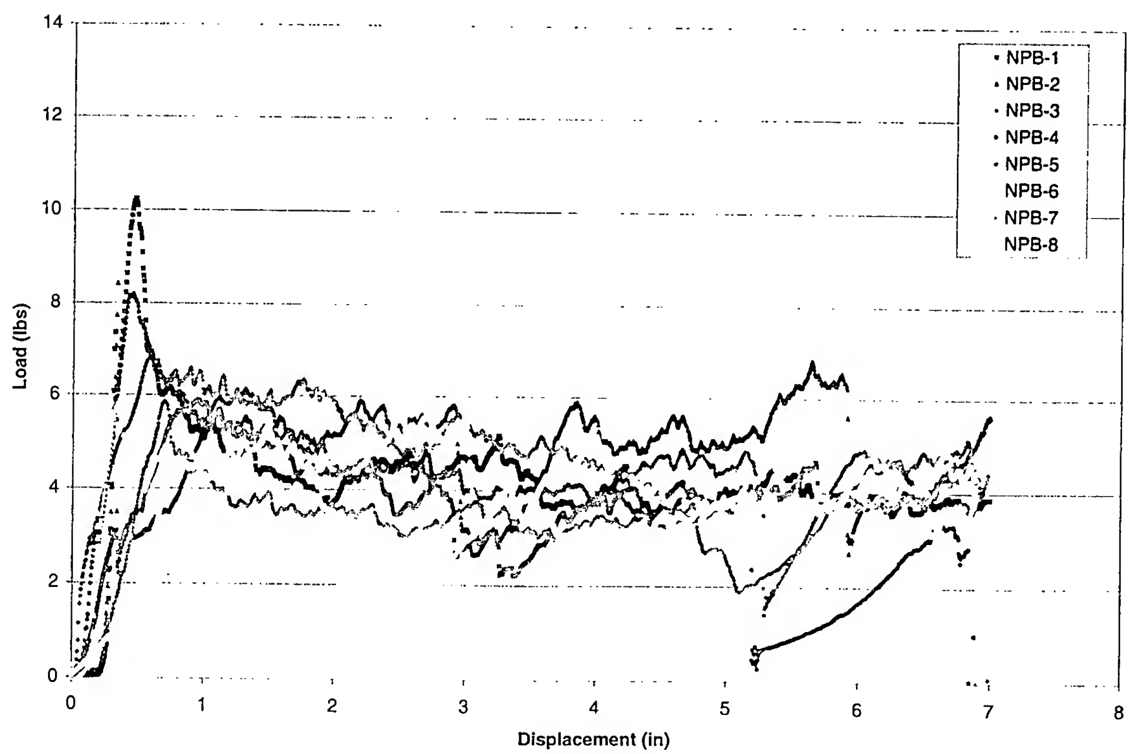

Figure B-4. NPB cleaned AM-355 (humidity exposed).

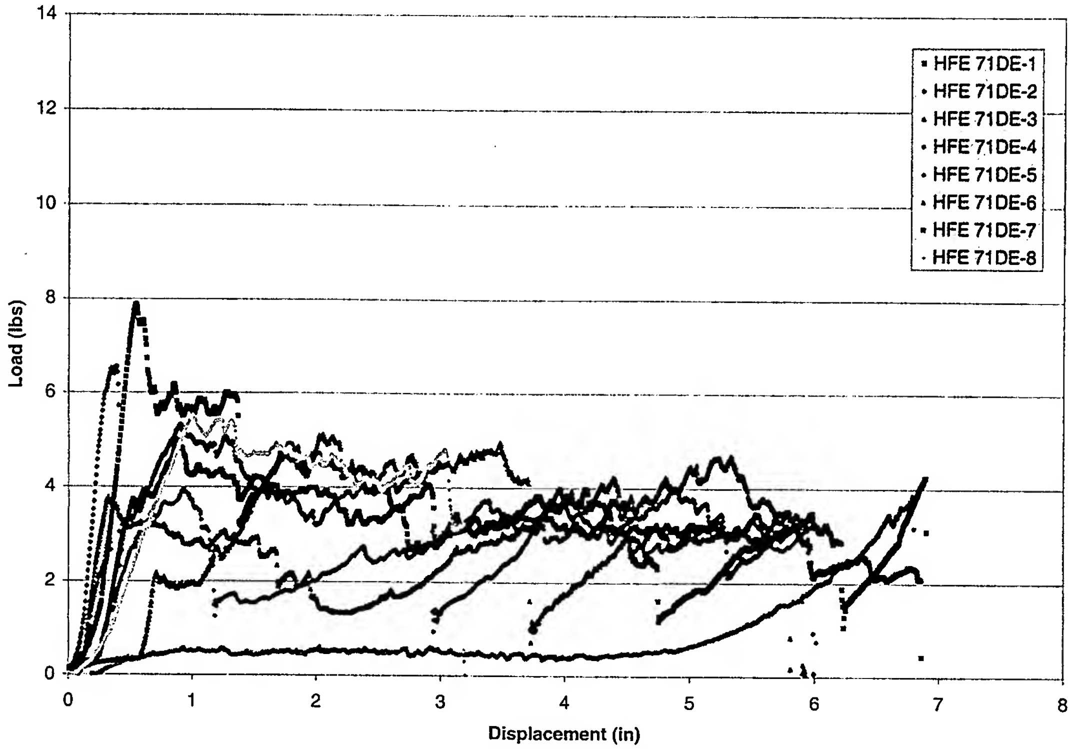

Figure B-5. HFE 71DE cleaned AM-355 (humidity exposed).

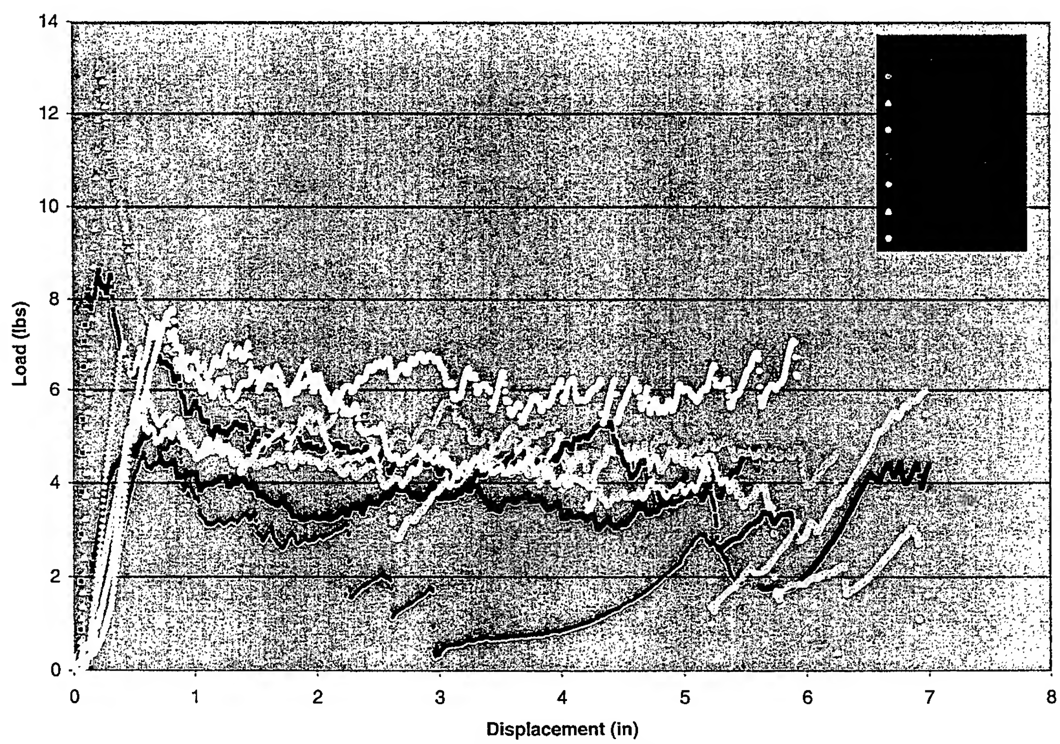

Figure B-6. Ethyl lactate cleaned AM-355 (humidity exposed).

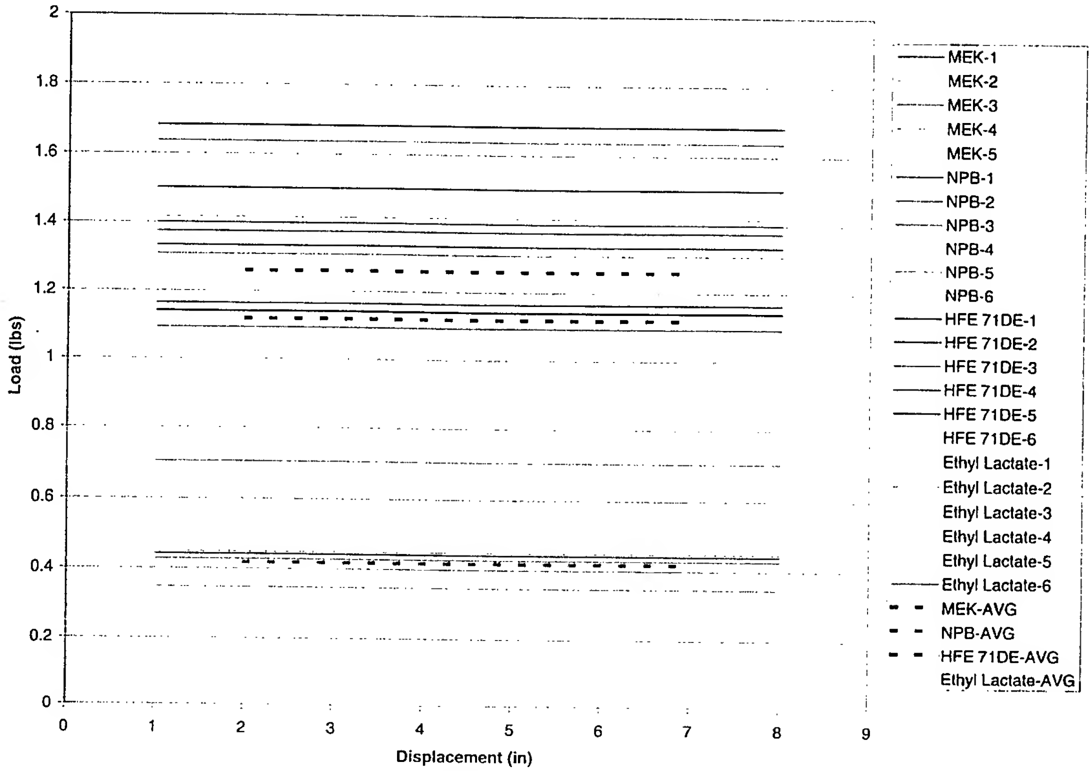

Figure B-7. Average AB load on electroformed nickel (humidity exposed).

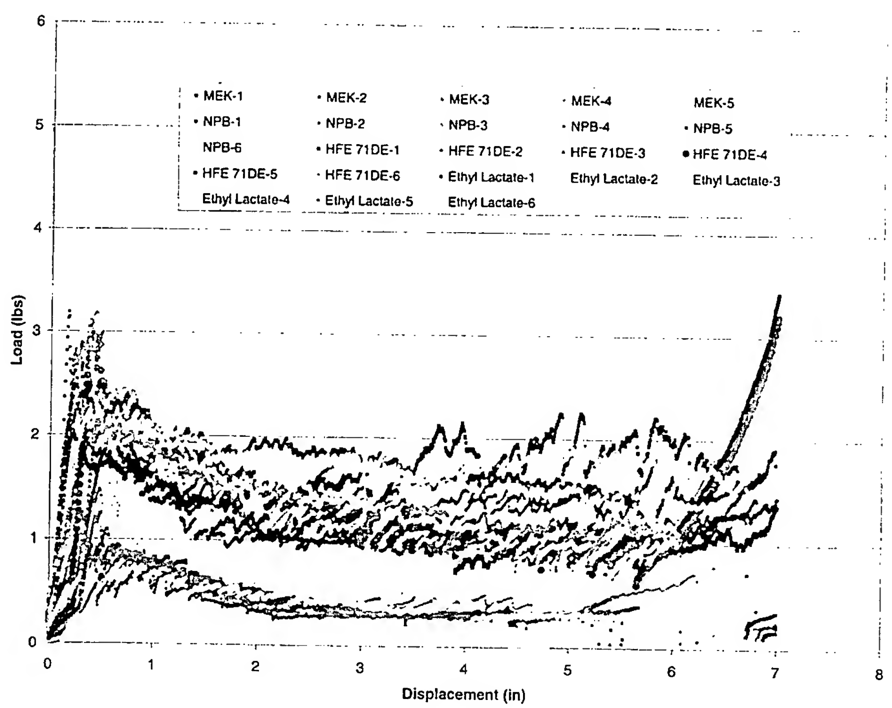

Figure B-8. Cleaned electroformed nickel (humidity exposed).

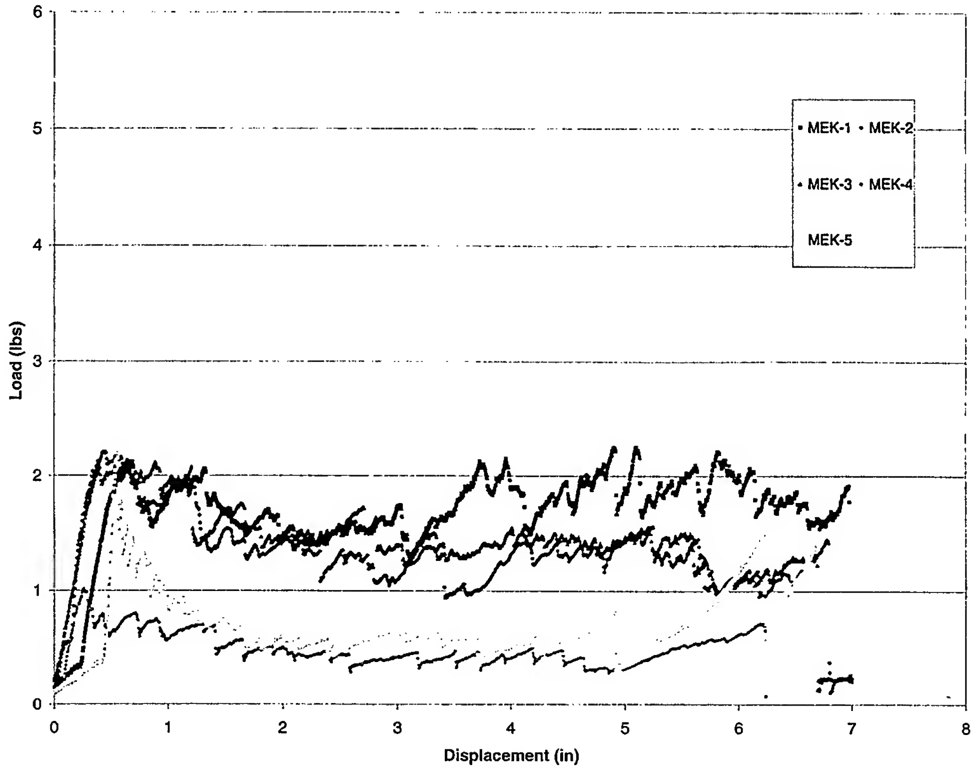

Figure B-9. MEK cleaned electroformed nickel (humidity exposed).

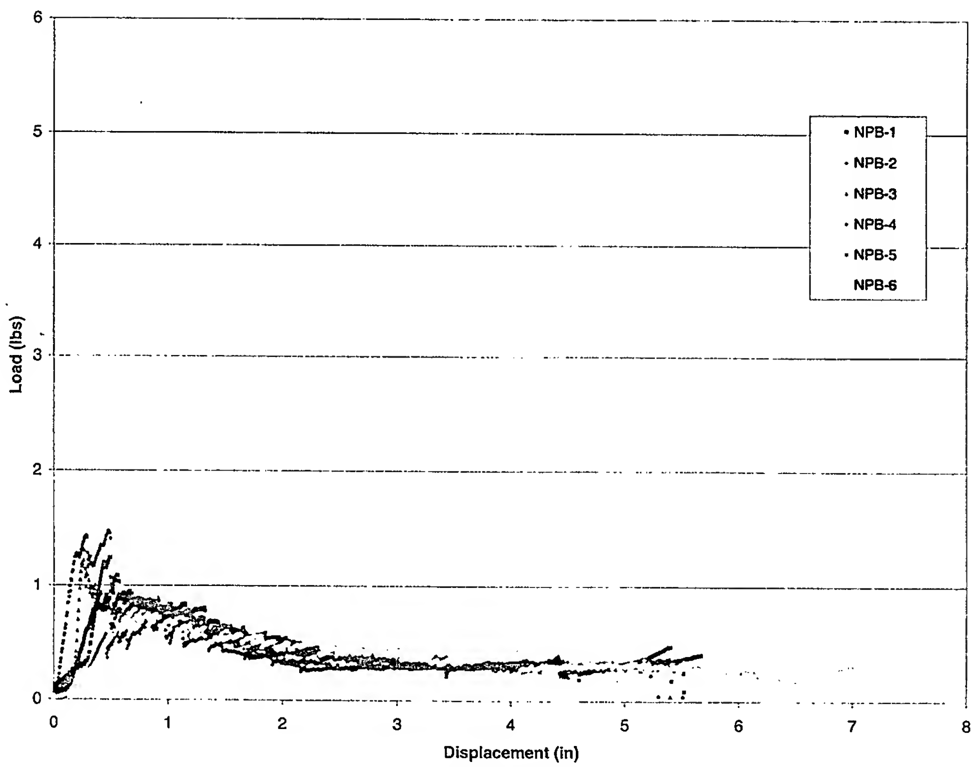

Figure B-10. NPB cleaned electroformed nickel (humidity exposed).

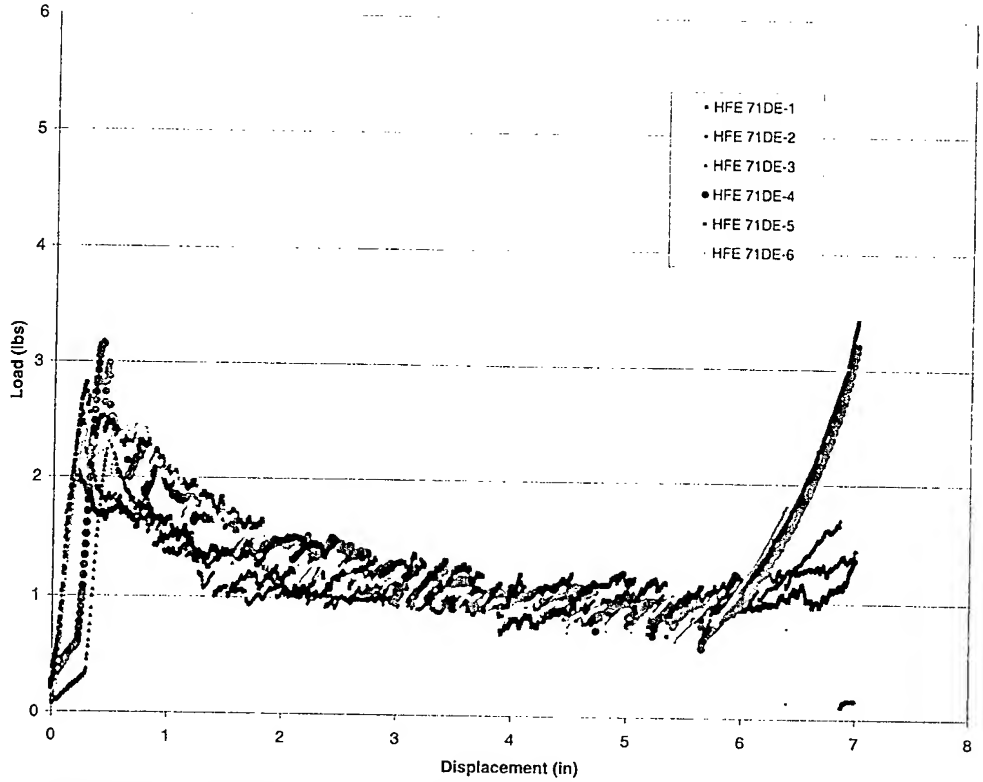

Figure B-11. HFE 71DE cleaned electroformed nickel (humidity exposed).

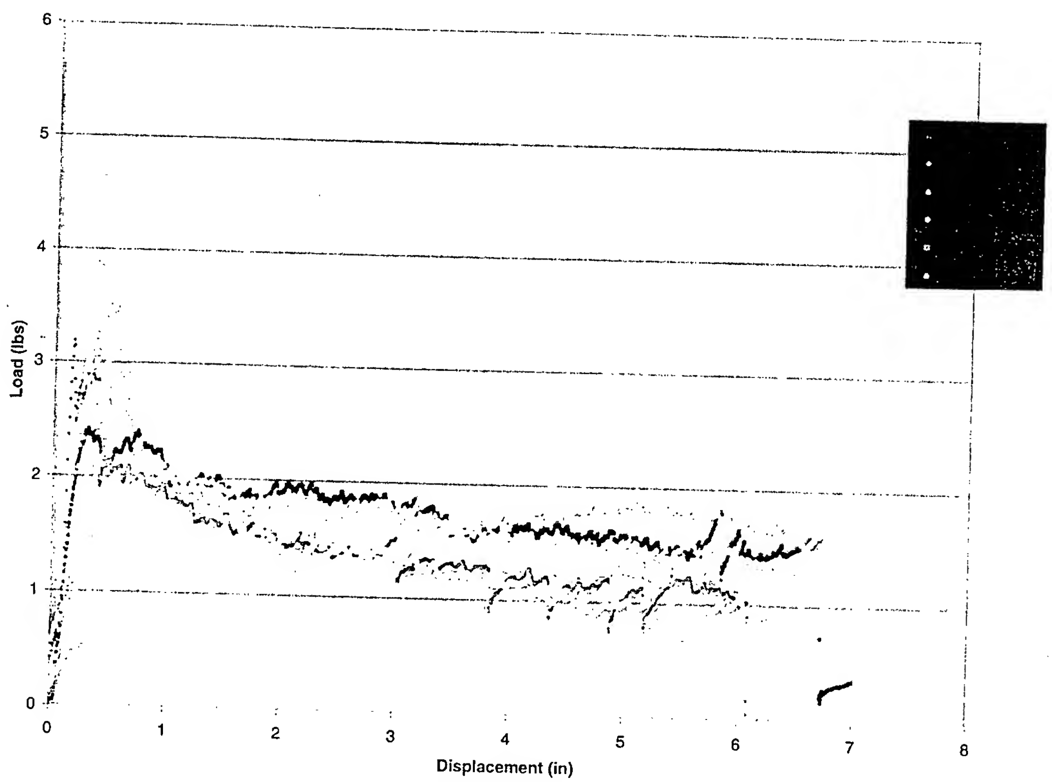

Figure B-12. Ethyl lactate cleaned electroformed nickel (humidity exposed).

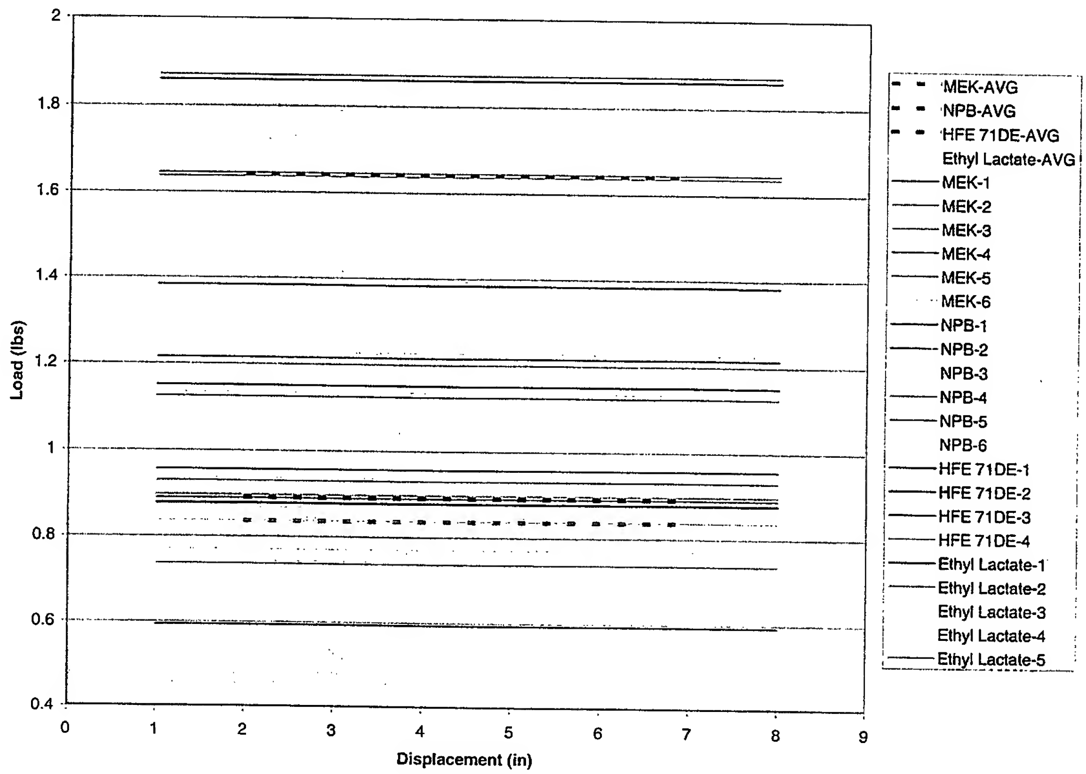

Figure B-13. Average AB load on titanium (humidity exposed).

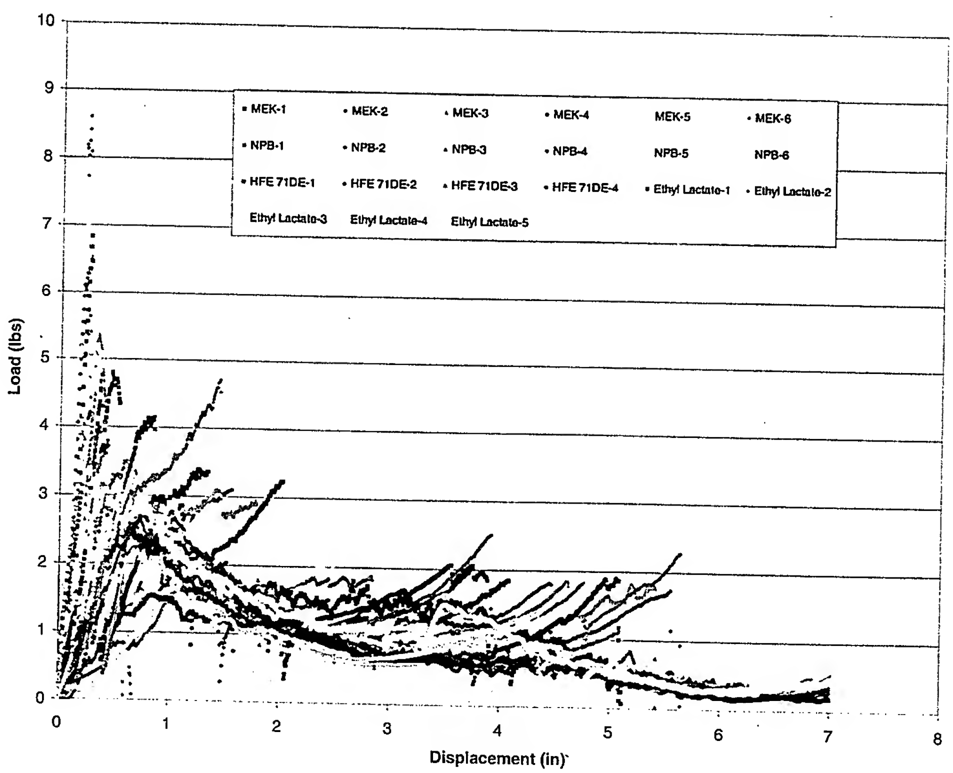

Figure B-14. Cleaned titanium (humidity exposed).

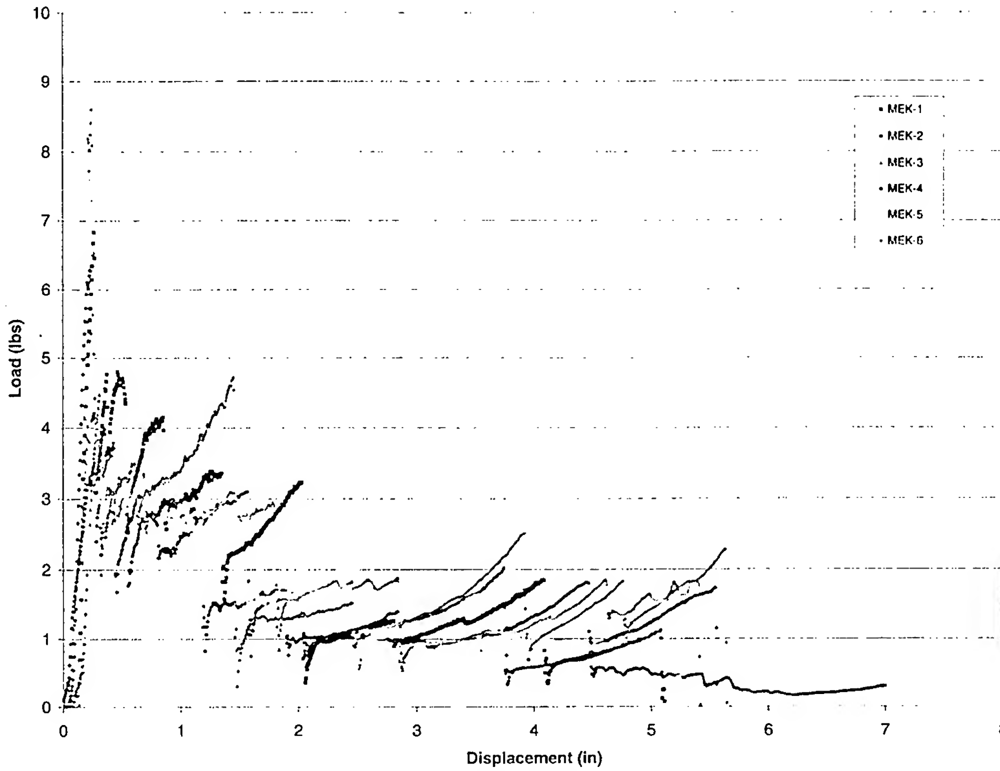

Figure B-15. MEK cleaned titanium (humidity exposed).

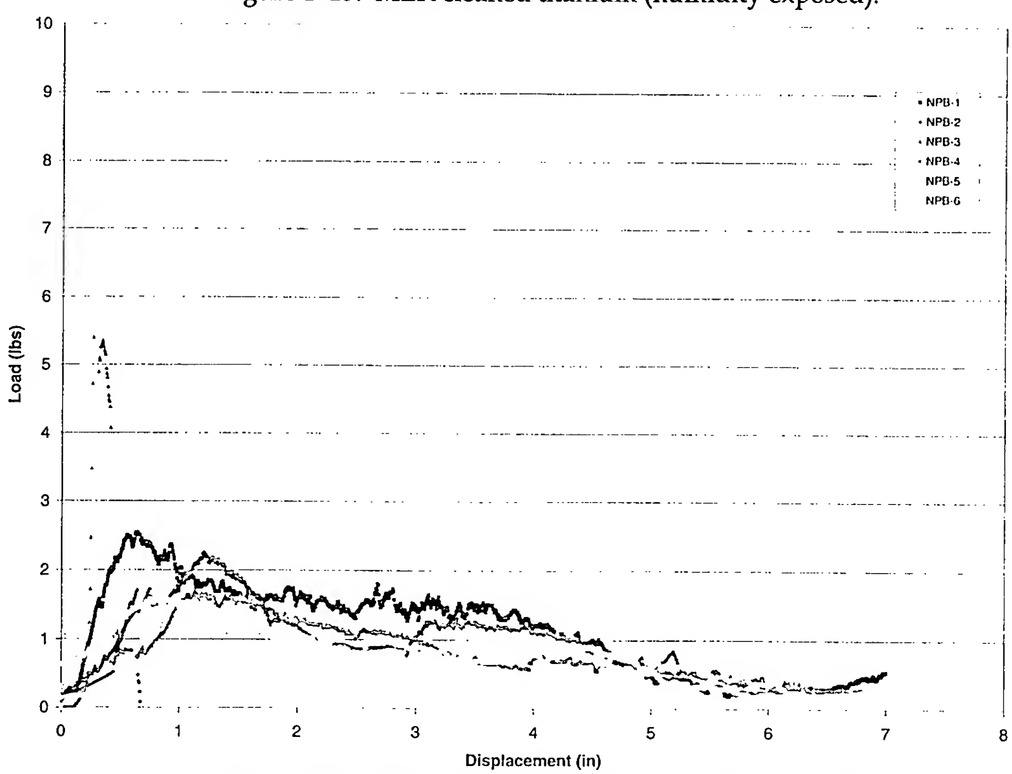

Figure B-16. NPB cleaned titanium (humidity exposed).

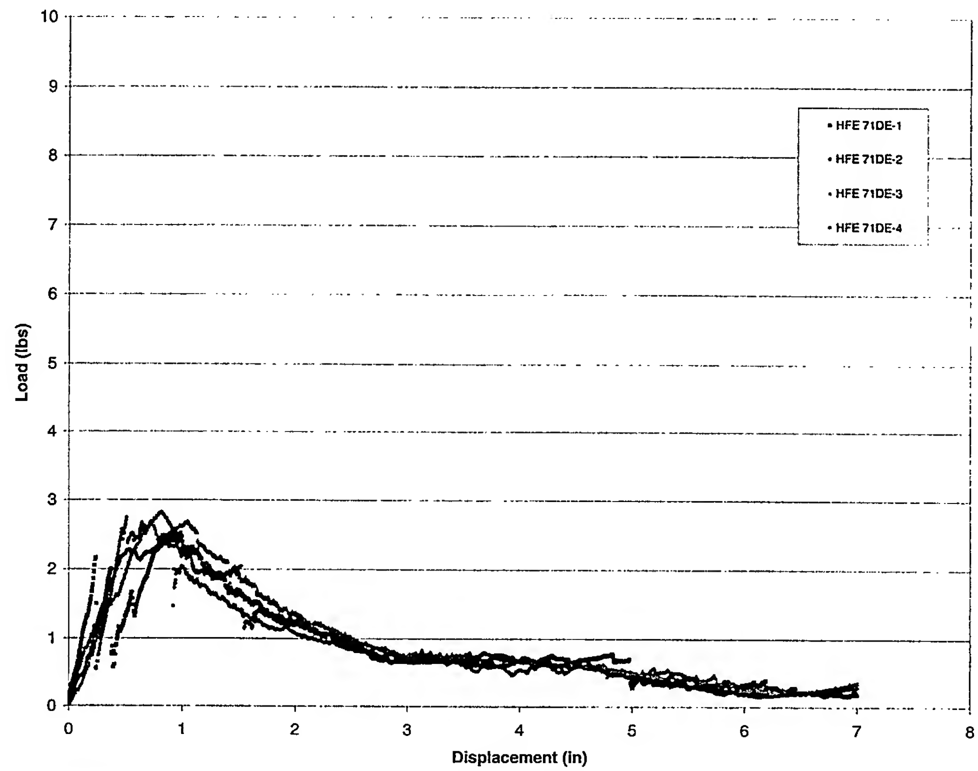

Figure B-17. HFE 71DE cleaned titanium (humidity exposed).

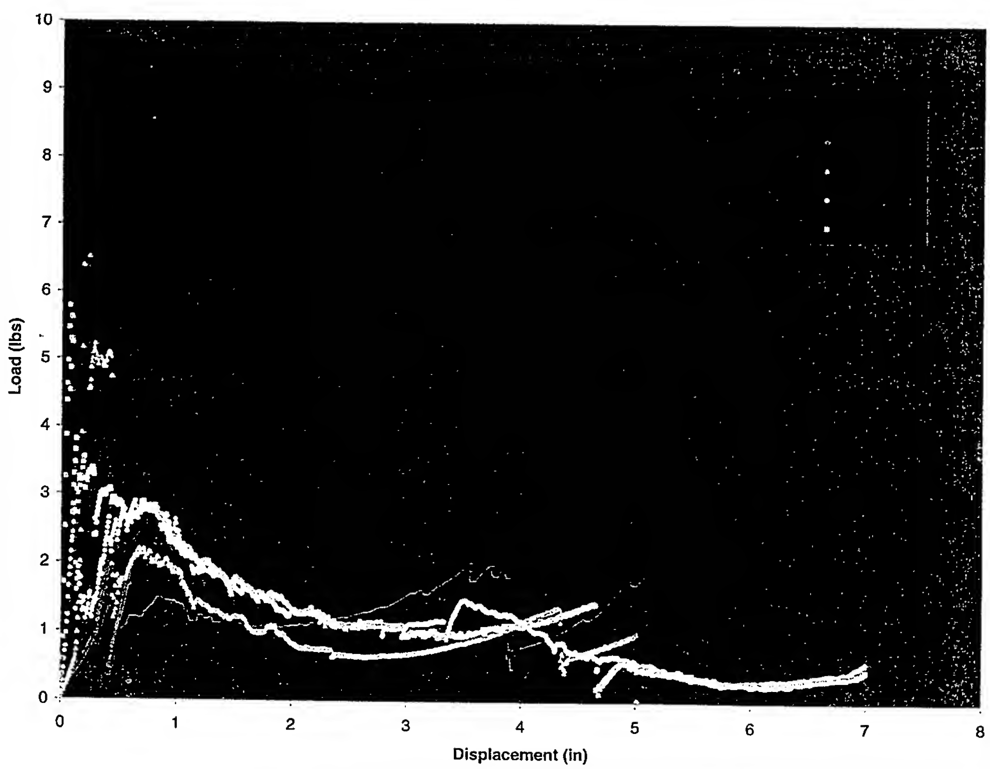

Figure B-18. Ethyl lactate cleaned titanium (humidity exposed).

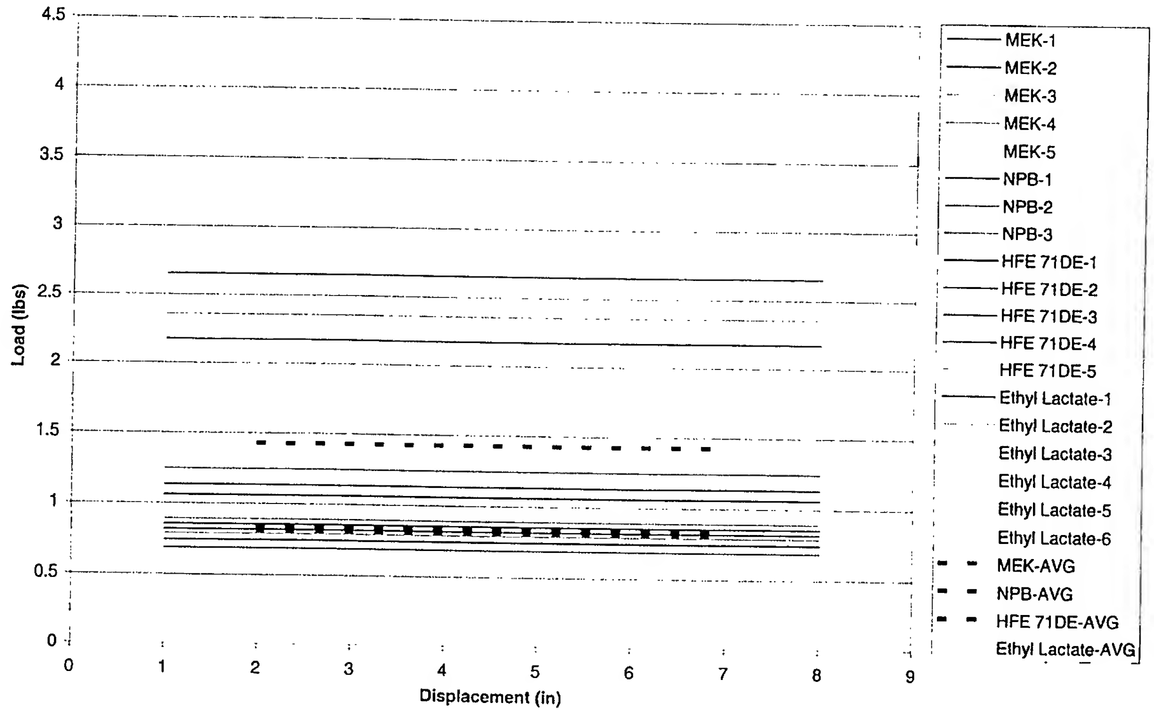

Figure B-19. Average AB load on aluminum (humidity exposed).

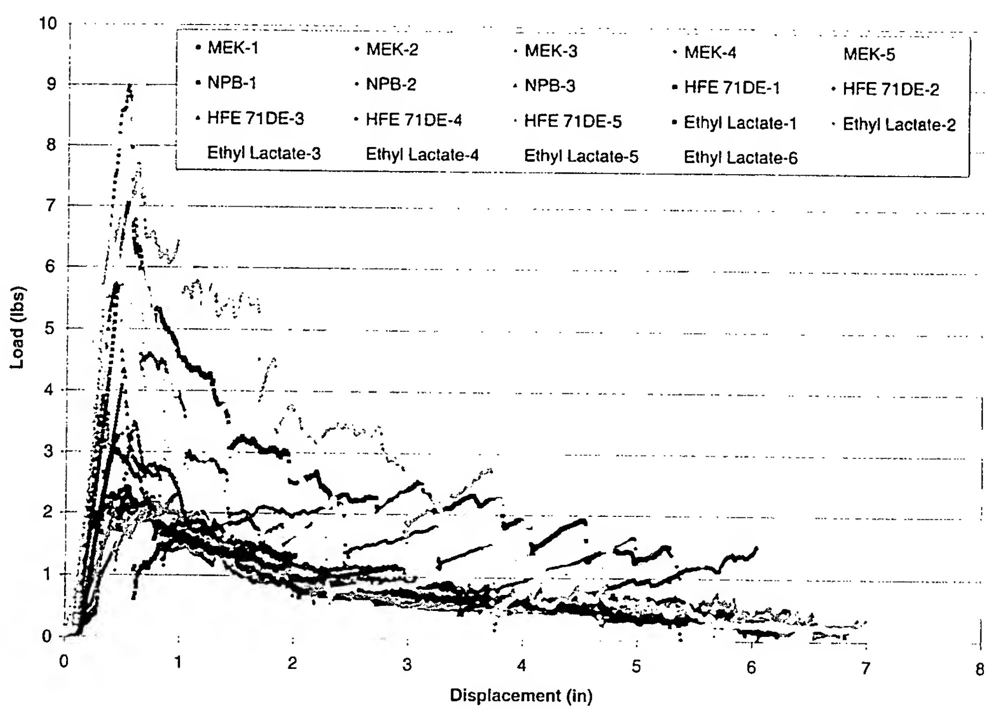

Figure B-20. AB performance of cleaned aluminum (humidity exposed).

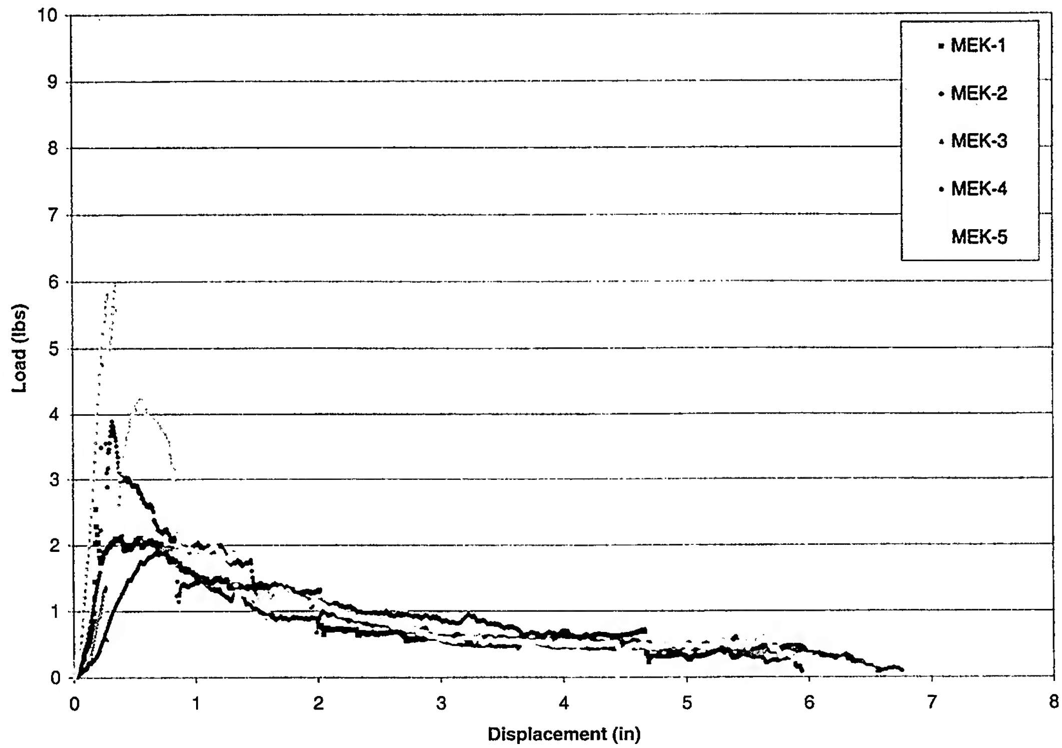

Figure B-21. MEK cleaned aluminum (humidity exposed).

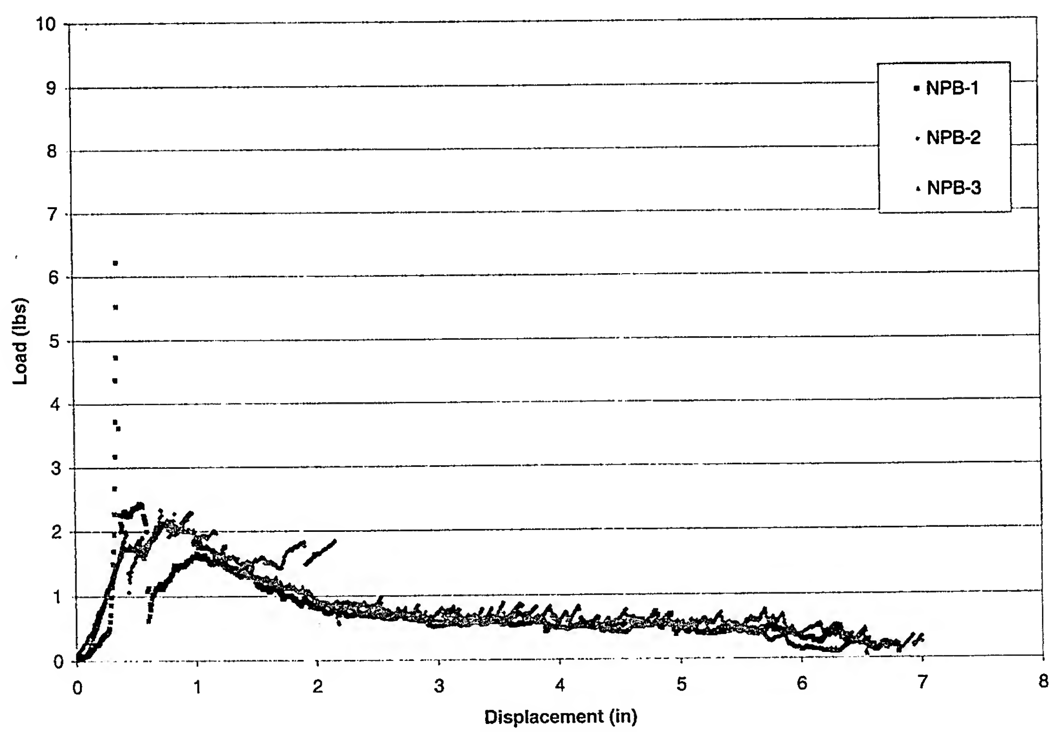

Figure B-22. NPB cleaned aluminum (humidity exposed).

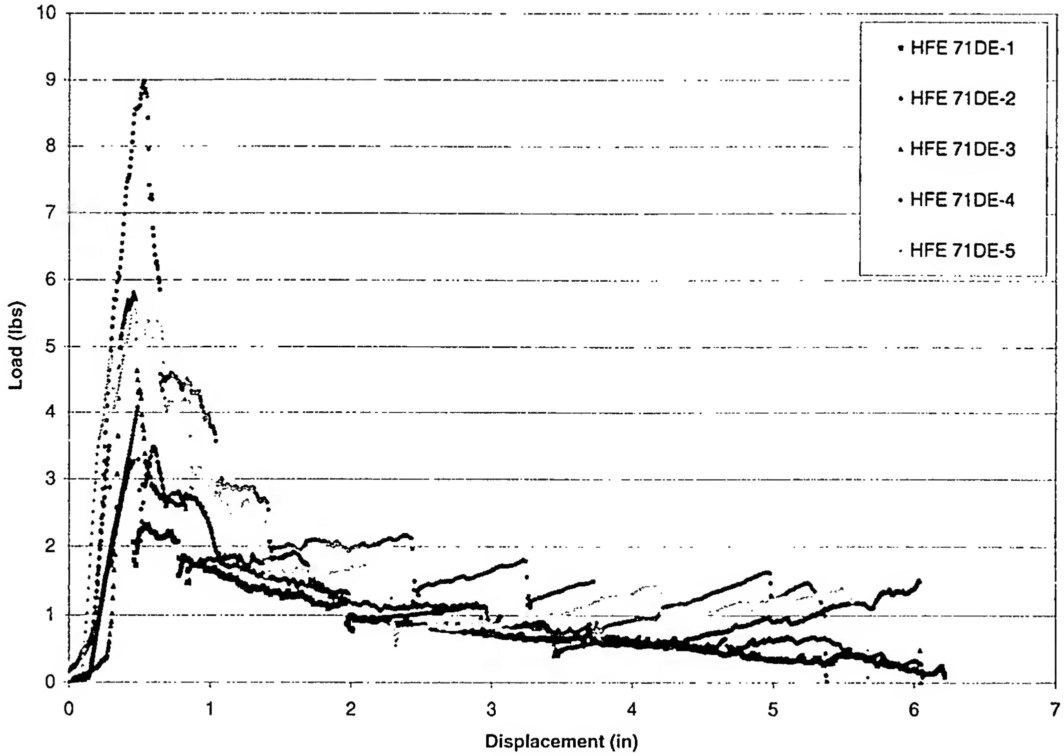

Figure B-23. HFE 71DE cleaned aluminum (humidity exposed).

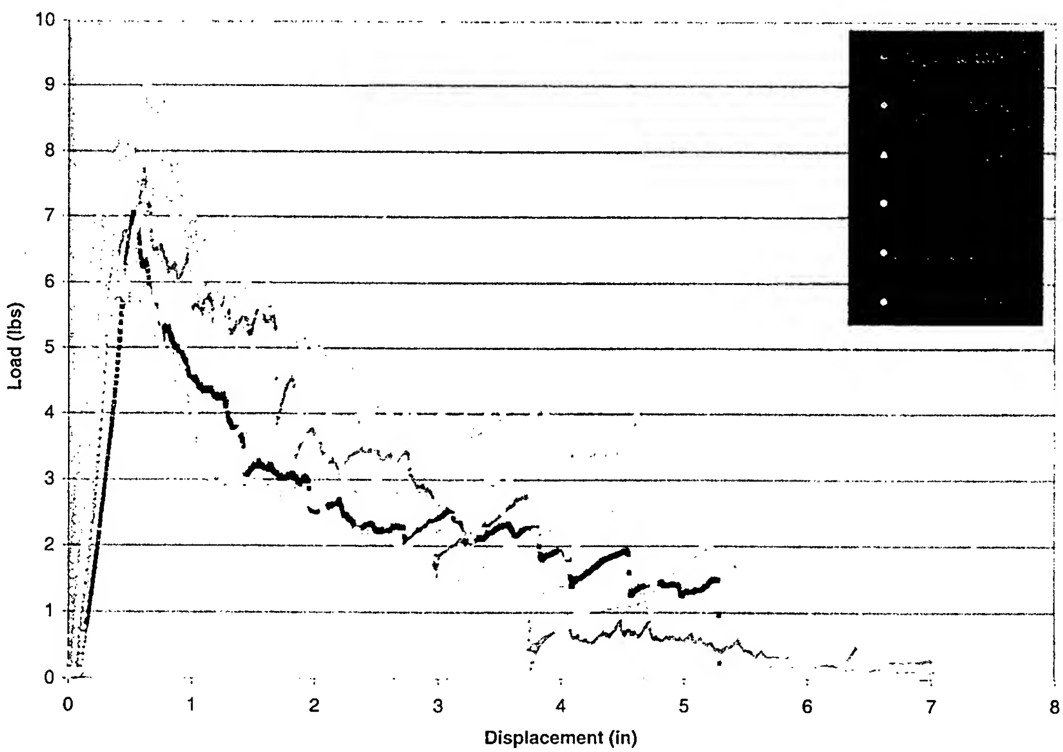

Figure B-24. Ethyl lactate cleaned aluminum (humidity exposed).

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13. ABSTRACT ***(Maximum 200 words)***

The purpose of this work was to identify a suitable, environmentally friendly maintenance chemical to replace methyl ethyl ketone (MEK), a standard solvent currently utilized for adhesive bonding of metal substrates. MEK was used as a baseline reference maintenance chemical for this project. The effectiveness of three environmental friendly replacement candidate compounds were evaluated under the repair simulation guidelines of the Aeronautical Design Standard Performance Specification for Cleaners, Aqueous and Solvent, for Army Aircraft (U.S. Army Aviation and Missile Command. "Aeronautical Design Standard Performance Specification for Cleaners, Aqueous and Solvent, for Army Aircraft." ADS-61-PRF, Draft, Aviation Engineering Directorate, Redstone Arsenal, AL, 16 May 2000), and the Standard Test Method for Floating Roller Peel Resistance of Adhesives, ASTM-D3167-93 (American Society for Testing and Materials. "Standard Test Method for Floating Roller Peel Resistance of Adhesives." ASTM D3167-93, West Conshohocken, PA, 1993). Four metal substrates (AM-355 stainless steel, electroformed nickel plated steel, aluminum 7075-T6 bare, and titanium 6Al-4V) and four chemicals (identified as MEK, normalized propylbromide [NPB], Vertec Gold, and HFE 71DE) were utilized. The adhesive utilized in the layup of the test specimens was the two-part epoxy paste system-Dexter Hysol EA 9309.3NA. The results indicated that the best replacement candidates were Vertec Gold, an ethyl lactate-based cleaner, and HFE 71DE, a solvent-based cleaner.

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